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DEVELOPMENT AND FABRICATION OF A GRAPHITE POLYIMIDE BOX BEAM FINAL REPORT

29 September 1972

Contract NAS8-24511, Modifications 4 Through 9

Prepared by

M. A. Nadler and F. J. Darms

Prepared for George C. Marshall Space Flight Center, Alabama 35812

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DEVELOPMENT AND FABRICATION OF A GRAPHITE POLYIMIDE BOX BEAM

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ABSTRACT

THIS REPORT DESCRIBES THE DEVELOPMENT, FABRICATION, AND THERMOSTRUCTURAL TEST OF A GRAPHITE FIBER REINFORCED POLYIMIDE COMPOSITE WING BOX BEAM, DESIGNED ON THE BASIS OF OV-10A GUIDELINE CRITERIA. MODMOR II/GEMON L WAS SELECTED AS THE COMPOSITE MATERIAL FOR REASONS OF AUTOCLAVING AND OVERALL BEST PROCESSING. THIS MATERIAL HAS A SERVICE TEMPERATURE OF 500 F. ALL ADHESIVE BONDING OPERATIONS UTILIZED FM-34 ADHESIVE. POSITIVE PRESSURE IN ASSEMBLY BONDING WAS APPLIED BY A SPECIAL TOOL, WITH VACUUM ASSIST TO REMOVE VOLATILES FROM THE GLUE LINE.

THE TEST ARTICLE WAS SUBJECTED TO COMBINED BENDING AND TORSIONAL LOADS WHILE EXPOSED TO 500 F. LOADS WERE APPLIED INCREMENTIALLY UNTIL FAILURE OCCURRED AT 80 PERCENT OF DESIGN LIMIT LOAD.

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FOREWORD

This final report covers the work performed on a graphite polyimide wing box beam during the period 1 July 1970 to 29 September 1972, on Contract NAS8-24511, Modifications 4 through 9. This program was sponsored by the National Aeronautics and Space Administration, George C. Marshall Space Flight Center, Alabama. Mr. E. L. Brown served as COR (contracting officer's representative) until succeeded by Mr. H. M. Walker in May 1972. The technical and physical aspects of the program were performed by the Space Division of North American Rockwell Corporation, with Messrs. M. E. Fields, L. J. Korb, and F. J. Darms serving successively as program managers. Mr. Darms also was responsible for structural design and testing. Other major contributions were made by Messrs. M. A. Nadler, materials and process development and assistance in preparation of the final report; J. S. Jones, specimen and structural fabrication and testing; and W. Wolstencroft and S. Y. Yoshimo, test article fabrication.

Publication of this report does not constitute NASA approval of the program results or conclusions. It is published only for the exchange and stimulation of ideas.

CONTENTS

ILLUSTRATIONS

TABLES

1.0 INTRODUCTION

The high strength-to-density and modulus-to-density ratios of advanced composite materials have led to numerous application studies of primary as well as secondary aerospace structures. A majority of these applications have been aircraft-oriented with maximum service temperatures below 350 F. For these applications, epoxy resins have served as the matrix material for boron and graphite fibers. Currently, sufficient flight test experience has been accumulated on boron/epoxy hardware to warrant classification as state-of-the-art for secondary aircraft structures. Although boron/epoxy and graphite/epoxy primary structures are undergoing flight and service tests, additional data are needed to achieve the confidence level necessary to make these applications state-of-the-art.

Aerospace structures operating in the service temperature regime of 500 F and 600 F recently have received attention in applications analyses of advanced composite materials. At the start of this program, advanced composites for this service temperature had been confined to small items that generally permitted the use of press and matched metal die molding processes. The reason for the lag between epoxy and high temperature material utilization was the complexity of the process required for the polyimide resins used as matrices of high temperature structural material systems. The development of addition-type polyimide resins and laboratory scale processing studies of other polyimides indicated that fabrication of large structures from these materials was feasible. These developments were of special interest for high temperature structures, where conventional metals such as titanium would be used because of service temperatures. This conceptual approach was taken in early versions of the Space Shuttle structure which minimized the thermal protection system by using titanium at service temperatures in the 500 F to 600 F range.

The advantages of fabricating and testing large structural components, often prior to complete development of the materials of construction, lies in the early development of all technical disciplines required to produce viable structures. By removing the restraint of laboratory size specimens from the problem, the technologies of manufacturing, design, and materials selection are required to interact. Therefore, problem solving and definition are placed on the scale of real structure. Processes are thus forced to comply with large component requirements. If the component can be directly compared to conventional material production hardware, a believable base for performance and cost estimates can be established.

This program investigated the application of graphite/polyimide (G/PI) materials to primary aerospace structure by the fabrication and test of a composite component to the requirements of the OV-10A aircraft center wing box beam. Similar idealized versions of the OV-10A center wing section have been used in a succession of previous North American Rockwell Columbus Division programs as a comparison test bed for defining the utility of various composite material systems in terms of manufacturability, performance, and special problems encountered. Material systems scrutinized in the past were glass-fabric/epoxy (References 1 and 2), boron/epoxy (Reference 3) and boron/polyimide (B/PI) (Reference 4). Test of the B/PI box beam had not been accomplished at the start date of this program.

Planning ground rules for this program assumed that: (1) The polyimide matrix resin selected for the B/PI program would probably prove optimum for the G/PI system; (2) adhesive assembly bonding of box beam members would follow an essentially identical approach; and (3) existing tooling would be adequate due to the expected configurational similarity of the B/PI and G/PI articles. None of the above assumptions proved viable as the program progressed for the following reasons:

1. The production material received for the B/PI program (boron fiber with DuPont-type 4707 resin) did not match the processing characteristics of previous lots used in orientation and development tests. A water soak cycle was employed in order to make it processable. This procedure is possible with B/PI prepreg which employs a glass-scrim carrier, but not with the unsupported G/PI. The process also was considered to be uncontrollable for production article utilization.

2. The B/PI box beam program employed 10 psi dead weight loading by means of a steel pressure distributing plate for effecting the major structural bonds between the cover panels and the spar/rib member with FM-34 adhesive. Failure of the B/PI article to meet design expectations (structural test failure at 83 percent of limit load) indicated that a change in the bonding approach is necessary.

The scope of the original program was amended during the study to include the process and tooling development necessary to produce a viable component representative of graphite/polyimide state-of-the-art. With these changes to the original approach, a center box beam was fabricated and tested.

2.0 SUMMARY

The purpose of this program was to evaluate the state-of-the-art of graphite/polyimide structures and to define key design and fabrication issues to be considered in future hardware programs. This objective was accomplished by the fabrication and test at 500 F of a graphite/polyimide center wing box beam using OV-10A aircraft criteria. The baseline design of this box was developed in a series of studies of other advanced composite materials - glass/epoxy, boron/epoxy, and boron/polyimide (References 1 through 4). The use of this basic design permits ready comparison of the performance of graphite/polyimide with these materials. Modifications to the baseline composite design were made only in those areas effected by the change of materials.

Processing studies of graphite fiber polyimide resins systems resulted in the selection of a Modmor II/Gemon L material. The fiber is a high strength graphite and the resin is an addition type polyimide with a 500 F service temperature capability. Modmor II was selected over HTS fiber when sporadic blistering was encountered with the latter. (This problem was caused by a surface treatment deficiency - since corrected by the fiber producer.) The Gemon L resin was selected over other candidates by reason of its superior processability. Design values for Modmor II/Gemon L were established on the basis of specimen tests for mechanical and thermophysical properties at room temperature and 500 F on unidirectional and crossplied laminates. The unidirection laminates were tested in the direction of and at 90 degrees to the fibers. The crossplied laminates were identical to the box beam cover skin laminates.

The graphite/polyimide composite center wing box fabricated and tested on this program is shown in Figure 2-1. It is 84 inches long, 38 inches wide, and 11.75 inches high. The structure is composed of upper and lower cover plates, two spars, and two ribs. Spars, ribs, and cover plates are precured and joined by secondary bonds using FM-34, a polyimide adhesive. Important structural features are listed below:

1. Cover plates — The cover plates were of a honeycomb sandwich construction. They utilized graphite/polyimide laminate skins and glass/polyimide honeycomb core. The skins were laminates of 12 plies of material laid-up in a 6/2/4 ratio in the spanwise/chordwise/45-degree directions. These laminates were approximately 0.063-in. thick. Upper (compression) and lower (tension) covers differed only in the thickness of the honeycomb core.

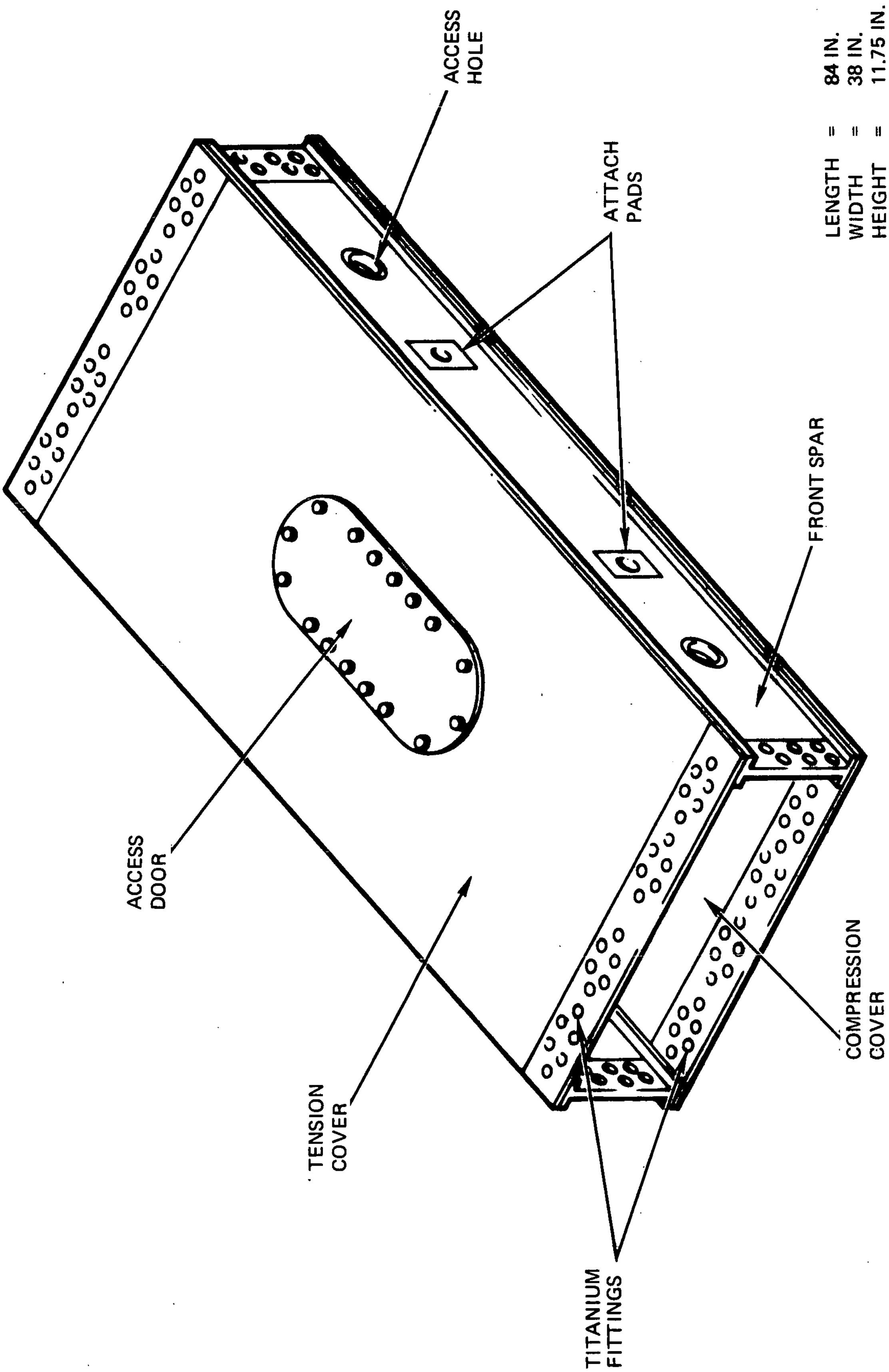

Figure 2-1. Graphite Polyimide Box Beam

2. Spars — The sparwebs were of honeycomb sandwich construction with face sheets composed of six layers of graphite/polyimide positioned at plus and minus 45 degrees to the span for maximum shear properties. These laminates were balanced across the honeycomb. Glass/polyimide honeycomb core was used in the sandwich. Glass/polyimide angles were bonded to the spar web to provide a bond surface for attachment of the spars to the cover plates.

3. Ribs — The honeycomb sandwich ribs were of the same type of construction as the spars with the exception that glass/polyimide was used for the skins. They are located immediately inboard of the attachment fittings.

4. Titanium fittings — Loads were applied to the cover plates and spars at each end of the beam. Mechanical attachment of the load application hardware to these components was accomplished by bolting through titanium end fittings incorporated into the honeycomb sandwich skins. These fittings were bonded to the skin in a stepped lap adhesive joint that was co-cured with the basic skin laminate. A solid section of glass/polyimide replaced the honeycomb at the position of the bolts.

5. Attachment Points — Loads were reacted at four attachment points - two in each spar. These points contained bearings which were located within solid, molded chopped glass/polyimide fittings. The fittings extended over the height of the spar.

6. Door — An access door was located in the center of the tension cover.

Adhesive bonding studies included co-curing of Modmor II/Gemon L with FM-34 for integral face sheet lamination/titanium end fitting bonding; secondary continuous surface bonding; and honeycomb core sandwich bonding.

Tests confirming major design features were performed on structural elements configured to represent the following details: 1) cover panel step lap joint between titanium end fittings and composite facings and 2) spar web including stepper lap titanium end fittings. These elements provided the data used to predict the ultimate strength of the box beam.

The article was fabricated utilizing autoclave techniques for producing the composite details of the rib, spar, and cover panel members, and for assembly of the individual members. The rib-to-spar sub-assembly and the final assembly bonds were carried out on a special positive pressure tool, with vacuum assist for volatile removal. This tool with vacuum bag assist permitted removal of volatiles from the FM-34 adhesive used for secondary bonds.

Quality control measures in the program included receiving inspection tests on incoming materials, tests on in-process control specimens, and tag ends fabricated simultaneously with the various operations, pre-fit checks by means of Verifilm 641 mismatch tape, sonic interrogation of joints, and visual and dimensional inspection of parts and the final assembly.

The box beam was static tested at 500 F under a combination of bending moment, shear, and torsion loads. Failure occurred at 80 percent of the design ultimate load predicted from coupon and structural element test data. This failure was the result of the rupture of a secondary bond of the spar to compression cover. It initiated at one attach pad and progressed by bond peel in both directions along the spar. An approximately vertical crack in the spar web, immediately inboard of the attachment pad, also was noted. This crack is believed to be a secondary failure resulting from de-bond of the spar and cover.

A review of the failure mode indicates that rupture was caused by the stress concentrations developed at the attachment pad hard point. The straight line deflection and strain data in the covers and spars indicate that these areas were sound. Therefore, it is believed that a design modification at the failure point would permit the achievement of the design ultimate load and that the manufacturing process used for the beam will produce viable structures.

Thirteen Technology Utilization items are credited to this graphite/polyimide box program. These items included co-cured laminate/adhesive bond processes, post-forming of glass/polyimide laminates, and felted paper bleeder material for molding composite laminates. Other program related new technology development efforts were performed to define a process and gather data as NASA-furnished Modmor II/polyquinoxaline.

3.0 COMPOSITE MATERIAL

3.1 MATERIAL SELECTION

Studies aimed at selecting the composite material for the G/PI Box Beam Program were a continuation of Company-sponsored development started several months earlier to select materials and develop processing and fabrication techniques for the application of G/PI composites to space vehicle structures. Table 3-1 summarizes the information generated in the IR&D program and available at contract commencement. From analysis of these and subsequent IR&D data, P13N (Ciba-Geigy), 4707 (DuPont), and Gemon L (General Electric) were identified as the three candidate resin matrix materials for final selection. In the majority of design studies at NR/SD on space vehicle structures, "high strength" graphite fiber reinforced composites had shown probable performance advantages over "high modulus" ones despite their lower modulus of elasticity. Overall design criteria were more efficiently met by these composites due to lower density, higher strength, and higher strain capability. This led to an a priori design decision for the G/PI Box Beam Program to limit graphite fiber choice to the "high strength" class, with Modmor II (Morgan-Whittaker) and HTS (Courtaulds-Hercules) being the contenders.

Resin Selection

Capability of the matrix resin to be cured by autoclave processes, with positive pressure not exceeding 90 psig and temperature not exceeding 450 F was established as a basic ground rule to permit hardware fabrication in existing NR facilities.

Other specific considerations leading to selection of the three candidate resins include:

P13N - The reaction mechanism of this addition-type polyimide resin, Figure 3-1, translates into reproducible low void structures with good interlaminar shear strength and associated resin dependent mechanical properties. Reportedly, articles of large dimensions may be prepared routinely because of the volatile-free cure mechanism. Since no post cure is required, the total processing time (in the order of two hours) is substantially shorter than those typical of condensation type PI's which generally call for lengthy involved post cures after pressure molding to develop maximum heat resistance. Disadvantages of P13N are extremely high heat rise rate requirements during

Table 3-1. Initial Status, Selection of PI/Resinfor Graphite/PI Composite

Fiber/PI Resin	Supplier	Cure Temp. (°F)	Press. (psi)	Post Cure Temp. (°F)	Horizontal Shear Strength (Ksi)			Flexural Strength (Ksi)				Flexural Modulus (Ksi)				Type of Cure Mechanism	Remarks
					R.T.	500	600	R.T.	500	600	650	R.T.	500	600	650		
Modmor II/P13N	WRD	550	300	None	12.6			274.8		155.5		18.8		18.4		Addition reaction	Not applicable to present autoclave equipment
A/P13N	Fiberite	550	300	None				114.8			87.8					Addition reaction	Not applicable to present autoclave equipment
S-Glass/P13N	WRD	550	300	None				193.9			118.4					Addition reaction	Not applicable to present autoclave equipment
Modmor II/4707	WRD	350	100	650	7.6		TBD	213.5		139.4*		21.0		TBD		Condensation reaction	Very touchy to process; cycling proc, staged
HTS Short/4707	Fiberite	350	100	650	5.5			177.6		81.4		16.5				Condensation reaction	Very touchy to process
HTS Short/4707	Fiberite	350	100	650	TBD		TBD	42.2*		TBD		TBD		TBD		Condensation reaction	Processibility improved by water soak
A Short/4707	Fiberite	350	100	650	4.2			148.3			61.9	16.5			11.7	Condensation reaction	Very touchy to process
S-Glass/4707	Fiberite	350	100	650	8.2			197.0			71.3	6.4			6.2	Condensation reaction	Very touchy to process
A/6234	Fiberite	350	100	650												Condensation reaction	Unsatisfactory process employed
Modmor II/703	WRD	350	100	650	TBD			TBD		TBD		TBD		TBD		Condensation reaction	In test
4T/Gemon-L	GE	350	100	600	TBD			TBD		TBD		TBD		TBD		Addition reaction	On order
S/S-RTS Cont. / Gemon-L	Hercules	350	100	600	TBD			TBD		TBD		TBD		TBD.		Addition reaction	1st panel being molded - bad batch, 2nd batch received - improved
HTS Short/Gemon-L	GE	350	100	482	10.6		5.6	181.1		169.9		18.6		22.3		Addition reaction	Excellent processibility
HTS Short/Gemon-L	GE	350	100	600	TBD		TBD	TBD		TBD		TBD		TBD		Addition reaction	Excellent processibility, panels blistered in P.C.
Modmor II/Gemon-L	GE	350	100	600	TBD		TBD	TBD		TBD		TBD		TBD		Addition reaction	4 panels in P.C. to 565 & 600 F
Modmor II/QX13	Fiberite	572	100	750	TBD		TBD	TBD		TBD	TBD	TBD		TBD		Condensation reaction	Material received, requires 572 F cure investigating lower temperature cure
RTS Short/QX13	Fiberite	525	100	750	TBD		TBD	TBD		TBD	TBD	TBD		TBD		Condensation reaction	Panel molded at 525 F appears ok; panel blister - P.C.
HTS Short/QX13	Fothergill & Harvey	572	100	750	TBD		TBD	TBD		TBD	TBD	TBD		TBD		Condensation reaction	Material received
Mod II/Rhone-Poulenc France (Kerimid 601)	Fothergill & Harvey	450	100	600	TBD		TBD	TBD		TBD	TBD	TBD		TBD		Condensation reaction	Material received
HTS/BPI 393	Brunswick	350	100	600	TBD		TBD	TBD		TBD	TBD	TBD		TBD		Condensation reaction	Material received

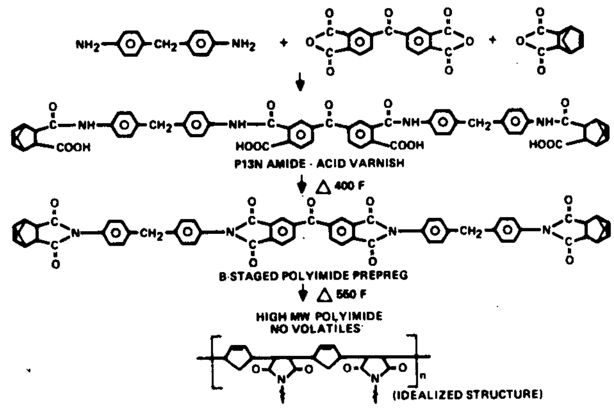

Figure 3-1. Reaction Schematic P13N

cure (not readily obtainable in conventional autoclave equipment), high molding temperature, and lower thermal stability than Monsanto and DuPont PI condensation systems. The latter are capable of long-term use at temperatures above 550 F, while 500 to 540 F is the range for long-term maximum maintenance of mechanical properties for P13N.

Pyralin 4707 is typical of PI materials produced by DuPont. They result from the condensation reaction of dianhydride and a diamine, as shown in Figure 3-2. The monomers in this particular example are oxydianiline and pyromellitic dianhydride. Water is liberated in the reaction.

4707 is the material selected and used in the Boron/PI Box Beam contract. The Graphite/PI program plan assumed a close relation between it and the Boron/PI, one with relatively easy transferability of the technology. Identical resin systems, if usable, would facilitate this transfer.

Comparison of analogous literature data indicated superiority of this resin over competitive condensation-polymerization type PI resins with respect to elevated temperature resistance.

Gemon L, produced by General Electric under agreements with Rhone Poulenc at contract inception[1] typifies another class of addition polymers as shown in the example of Figure 3-3.

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[1] This agreement is now terminated and General Electric has discontinued the pertinent product line including Gemon L. The related Rhone-Ponlenc resin, Kermid 601, is available through a U.S. distributor, Rhodia Corp, New York City, N.Y.

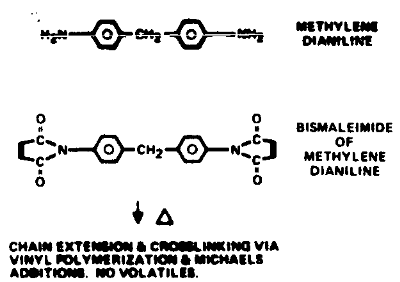


Figure 3-2. Reaction Schematic Rhone Poulenc Polyimide

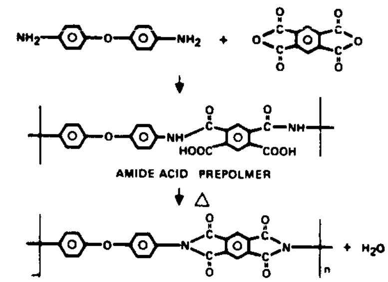


Figure 3-3. Reaction Schematic DuPont Polyimide

These materials are the reaction products of certain N, N'-bis-imides of unsaturated dicarboxylic acids with diamines. The resin technology in this field appears to be based on monomers containing a maleimide group rather than aromatic polymers. Thus alipliatic bridges or chains are created during cure in lieu of the aromatic polyimide chain extension of the other polyimides. Consequently, the thermal stability of this resin class is much lower. During oxidative or thermal degradation these aliphatic bridges undergo scission yielding the monomers which readily degrade or sublime. However, a major advantage is producibility under autoclave conditions at 350 F, with reproducible low void content and high mechanical properties.

The selection criteria established for selection of the resin matrix were the following:

1. Processability (capability of being autoclave-cured in conventional equipment).

2. Laminate soundness (indices: short beam shear strength and void content).

3. Maintenance of soundness after repeated temperature cycling (index: freedom from microcracking tendencies).

Studies on these criteria, leading to selection of Gemon L for the program, are described below.

Processability

The processes used in these investigations are described in detail in Appendix A. The starting points were vendors' recommended cure cycles, Process No. 1 for Pyralin 4707, No. 4 for Gemon L, and No. 9 for P13N.

Pyralin 4707. Processes 2 and 3, which were explored in addition to the "standard" Process No. 1, are variations of then proprietary, but now disclosed, methods developed at NR/Tulsa Division. They had been found effective in upgrading the product quality and repeatability of glass-fabric/4707 laminates.

As shown in Tables 3-2 and 3-3, the standard type processing cycle (Panel PD 30-1-6) did not produce satisfactory results. Reasonable results were obtained with cure cycles employing pressure cycling and with water soak procedures (Panels PD 26-1-10 and PD 31-1-13). Although water soak procedures were planned for the boron/PI box beam, far greater handling difficulties were anticipated for graphite goods which do not employ the glass fabric carrier used with the boron prepreg. None of the processes used was considered to be sufficiently reliable to warrant the risk of committing component fabrication to them and 4707 was dropped from further consideration.

P13N. The addition reaction type PI resin P13N exhibited, as expected, the highest properties both at room as well as elevated temperatures (Tables 3-2 and 3-3). However, no encouragement was received from the vendor and/or by study of the literature that this material, as constituted, could be made amenable to cure cycles which are compatible with existing tooling and equipment. It, too, was dropped from further consideration.

Gemon L. Orientation runs on Gemon L/graphite panels under IR&D, using the original cure cycle suggested by the vendor (Appendix A, Process No. 4) did not produce completely satisfactory results in all respects. Panel blistering was noted in the 600 F post-cure; fiber volume contents were 75.9% and 73.09% in the two panels made, in lieu of the target 60 ± 5%; void volumes were 8.6 and 4.9%, respectively; horizontal shear test specimens extracted from sound portions of the second panel resulted in average ultimate shear strengths of 10.6 Ksi at 600 F. The latter determination, however, indicated substantial inelastic behavior.

Discussions with the vendor, G.E., regarding improvement potential led to the following decisions:

1. Future use of a different solvent which was considered more suitable from the processing standpoint and with respect to elevated temperature resistance of fabricated laminates. The shipment of prepreg received subsequent to this discussion, also used for panels in the resin selection studies, was coded Batch HC 195, Resin Type M33B, with 2MA solvent.

2. A different curing schedule was suggested.

Panels were fabricated in the next orientation step by five processes, Nos. 4 through 8. All panels appeared sound, including the one fabricated by the schedule originally recommended by G. E. This supports the tentative conclusion that solvent substitution was helpful.

However, imprints from the glass fabric used between the restraining steel plates were noted on all panels, including the ones utilizing only a 565 F maximum post-cure temperature. This in itself indicated that the service temperature ceiling for Gemon L may have to be lowered even below that figure.

Furthermore, none of the data obtained, Tables 3-2 and 3-3, were attractive from the standpoint of retention of mechanical properties at the two elevated temperature levels tested, 565 F and 600 F. Also, the void contents found were considerably higher than expected, indicating a quality short of that which should be attainable and which was the principal reason for selecting the addition type Gemon L as a candidate. The general processing characteristics of Gemon L, however, were found so superior that additional improvement attempts were made.

A visit to G. E. and ensuing discussions elicited the belief that a fast post-cure schedule might be the cause for the disappointingly high void content observed. The relatively fast post-cure schedule did not permit residual volatiles to diffuse out. Slower post-cure cycles were used in subsequent improvement attempts.

Results of the tests performed are shown in Table 3-4. The fabrication processes referred to are described in Appendix A. It is evident that beneficial results were produced in terms of RT short beam shear strength and void content. The latter is below 1 percent, extremely good for a low pressure PI/Graphite composite. The fact that weight losses and lateral shrinkage during post-cure also are substantially lower than those observed in the preceding series is additional evidence for the soundness of lengthening the post-cure cycle. Since the reduction in initial volatile content may have been a contributing factor, this parameter was carried into the final resin selection studies.

Laminate Soundness

With selection of Gemon L for the remainder of the program on the basis of the processability studies, it was expected that the intended service temperature and related testing efforts would have to be reduced substantially below the originally projected 600 F level, with the preliminary expectation being 500 F. To verify the use temperature and check the effect of volatile content in the prepreg on laminate properties, the following experimental series was set up.

Table 3-2. Physical and Flexural Composite Properties Processability Studies

	Resin Type	4707			Gemon L					P13N
	Panel Designation	30-1-6	PD 26-1-10	PD 31-1-13	PG 11-2-19	PG 11-1-19	PG 10-2-19	PG 10-1-19	PG 12-1-21	P 12
	Tack	N.G.		Good	N. G.					Good
Prepreg	Flow, % (1)	21.0		16.09	15.31					20.34
	Volatiles, % (2)	17.93		24.57	6.3/10.31*					19.81
	Res solids, wt % (3)	35.71		30.81	34.0/34.61*					29.84
	Unit wt, gm./sq in.	0.2384			0.475					
	Fiber wt, gm./sq in.	0.1376		0.1153	0.0788					
	Nom fiber count Tows/in.				3.5					
Laminate	Flex str, ksi	No test; panel delaminated								
	RT		229.0	221.0	217.5	161.0	219.0	254.0	96.8	301.2
	565 F					19.4		62.1		
	600 F		123.4	129.0	19.8		37.0		21.0	162.3
	Flex mod 10^6 psi									
	RT		22.7	20.1	20.7	19.4	19.96	19.1	17.3	19.7
	565 F					2.4		9.8		
	600 F		19.8	21.0	2.4		4.33		2.2	18.2
	Density, lb/cu in.		0.0541	0.0542	0.0530	0.0538	0.0539	0.0540	0.0497	0.0567
	Fiber content, v/o (4)		65.1	73.7	69.4	68.1	66.8	67.7	69.1	60.0
	Thickness/ply mils		7.8	6.5	5.1	5.1	4.9	5.0	5.8	7.3
	Wt loss in PC %		3.6		4.8 at 600 F	4.9 at 565 F	1.3 at 565 F	2.2 at 600 F	6.4 at 600 F	N.A.
	Transverse shrinkage, PC %		1.7		1.2 at 600 F	1.7 at 565 F	0.6 at 565 F	0.8 at 600 F	1.9 at 600 F	N.A.
	Void content v/o		7.94	9.6	8.7	7.1	6.7	6.5	13.8	1.40

*Vendor/NR Findings

(1) Determined at 100 psi, 350 F.
(2) Determined at 750 F for 1 hour.
(3) Determined by boiling HNO_3 extraction, 215 F for 2-1/2 hours.
(4) Determined from density and boiling HNO_3 extraction measurements.

Table 3-3. Initial Short Beam Shear Strength Results
Processing/Resin Selection Studies

Material System and Process	Panel Design.	Shear Strength, Ksi			Remarks
		RT	565 F	600 F	
4707/Modmor II (40%) Process No. 1 (Std.)	PD 30-1-6	No Test			Panel delaminated
4707/Modmor II (40%) Process No. 2 (Pressure cycling)	PD 26-1-10	8.7 9.6 9.1 8.7 9.0 Av	5.9 5.9 6.2 6.0 Av	4.9 4.9 4.2 5.0 4.7 Av	All shear failures
4707/Modmor II (55%) Process No. 3 ("water soak")	PD 31-1-13	6.8 7.2 8.0 7.3 Av	6.1 6.2 6.5 6.2 6.3 Av	 4.6 4.5 4.5 Av	All shear failures
Gemon L/Modmor II Process No. 4 ("G. E. standard" plus 600 F P. C.)	PG 11-2-19	6.2 6.4 6.4 7.5 6.6 Av		Excessive deformation	
Gemon L/Modmor II Process No. 5 ("G. E. standard" plus 565 F P. C.)	PG 11-1-19	8.2 8.3 7.0 8.2 7.9 Av	1.6(Y) 1.6(Y) 1.7(Y) 1.6(Y) Av		
Gemon L/Modmor II Process No. 6 (No intermediate hold in cure; 600 F P. C.)	PG 10-2-19	7.2 8.2 7.9 8.3 7.9 Av		3.3(Y) 3.4(Y) 2.3(Y) 1.9(Y) 2.7(Y) Av	
Gemon L/Modmor II Process No. 7 (No intermediate hold in cure; 565 F P. C.)	PG 10-1-19	6.5 7.1 7.5 7.4 7.1 Av	4.2(Y) 4.2(Y) 3.9(Y) 4.0(Y) Av		
Gemon L/Modmor II Process No. 8 (Incremental Lay-up; 600 F P. C.)	PG 12-1-21	6.7 5.4 5.3 5.8 Av		1.3(Y) 1.2(Y) 1.3(Y) 1.3(Y) Av	
P13N/Modmor II Process No. 9 (575 F, 500 psi)	P-12	13.3 14.7 13.2 13.0 13.7 Av	7.4 7.6 7.4 7.5 Av	6.6 5.8 6.5 6.6 6.4 Av	All shear failures
(Y) denotes yielding failure, 0.2% offset					

Table 3-4. Slow Post Cure Processing Studies, Gemon L/HTS
Batch 7346-37-1

Panel design	PG 14-1-19	PG 14-2-22	PG 14-1-19	PG 15-2-22
Process No.	11	12	13	14
Flex strength, ksi				
RT	209.8	195.0	216.6	209.7
565 F	65.0	60.0	66.5	73.6
Flex mod, 10^6 psi				
RT	18.0	18.5	18.9	18.4
565 F	11.3	9.6	12.3	14.6
SBS strength, ksi				
RT	12.3	13.3	11.4	13.4
500 F			6.0	5.0
565F	4.1	3.5	4.3	4.6
Density, lb/cu in.	0.0562	0.0560	0.0563	0.0558
Fiber content, $v/_o$	56.8	56.3	57.5	54.6
Thickness/ply, mils	5.6	5.7	5.7	5.4
Wt loss in P.C., %	0.8	1.2	0.5	0.7
Transv shringage in P.C., %	0.6	0.9	0.3	0.4
Void content, $v/_o$	0.42	0.59	0.60	0.51

Material to be obtained from G. E. to nominal volatile contents of 10, 8, 6, and 4 percent. Panels to be prepared from each of these prepregs per Process 15 (Appendix A), using slow post-cures to 500 F, 565 F and 600 F, repectively. Short beam shear specimens were to be prepared from these panels and tested at RT, 500 F, 565 F, and 600 F.

Gemon L/continuous HTS material, ordered to volatile contents of 10, 8, 6, and 4 percent was received from General Electric. Close checking revealed some differences in G. E. and NR/SD test methods for volatiles. As a result, the parameters for volatile determination were changed from

750 F for one hour to 600 F for 15 minutes to insure a valid comparison of results. The 750 F determination is not satisfactory since the Gemon L resin is not stable at that temperature. Inspection tests performed with the new parameters on different portions of the 30-inch wide, 36-inch long sheets revealed considerable discrepancies from the stipulated requirements as well as nonuniformity, see Table 3-5.

Despite these discrepancies it was decided not to subject the program to any delays incumbent on material rejection, since the major purposes of the projected short beam shear test series could still be accomplished with the received shipment: i. e., (1) The existing data base for resin selection could be extended and confirmed for the Gemon L resin system utilizing 2MA solvent (the previous series had contained a dioxane solvent), (2) the desired prepreg volatile percentage could be ascertained and documented, and (3) the processing parameters could be verified. The vendor, General Electric, was notified that future deviations from procurement stipulations would not be tolerated, unless not considered to affect the program.

Panels (6.1 in. by 4.25 in. by 14 ply) for short beam shear tests were prepared from each of the four materials received using Process 15 (Appendix A). The panels were cut into three equal pieces which received post cures extending to 500 F, 565 F, and 600 F, respectively. This process is similar to Process 14, except it provides for post-cure maximum temperature options and a longer hold at the maximum post-cure temperature.

Results of the short beam shear tests at RT and 500 F are presented in Table 3-6. Tests had been planned at RT, 500 F, 565 F, and 600 F for specimens representative of all the above processing conditions. Microscopic examination of the specimen edges revealed that the post cures exceeding 500 F had produced definite interlaminar splitting with evidence of resin cracking as shown in the 40X photomicrographs, Figure 3-4. Despite relatively high short beam shear strength of laminates post cured at 565 F and 600 F, Table 3-3, the splitting tendency is sufficient evidence of service unreliability at these temperatures to cause their rejection. A firm decision was made to use 500 F as the elevated temperature baseline for this program and restrict testing efforts to this baseline.

A noteworthy observation made in the RT short beam shear tests was that many specimens failed in fiber tension as shown in Figure 3-5. This is the first time this failure mode had been experienced at NR/SD on a graphite/PI system tested at a 4:1 span-to-depth ratio and is indicative of excellent fiber-to-resin bonding.

Table 3-5. Physical Properties of Gemon L/HTS Prepreg(1)

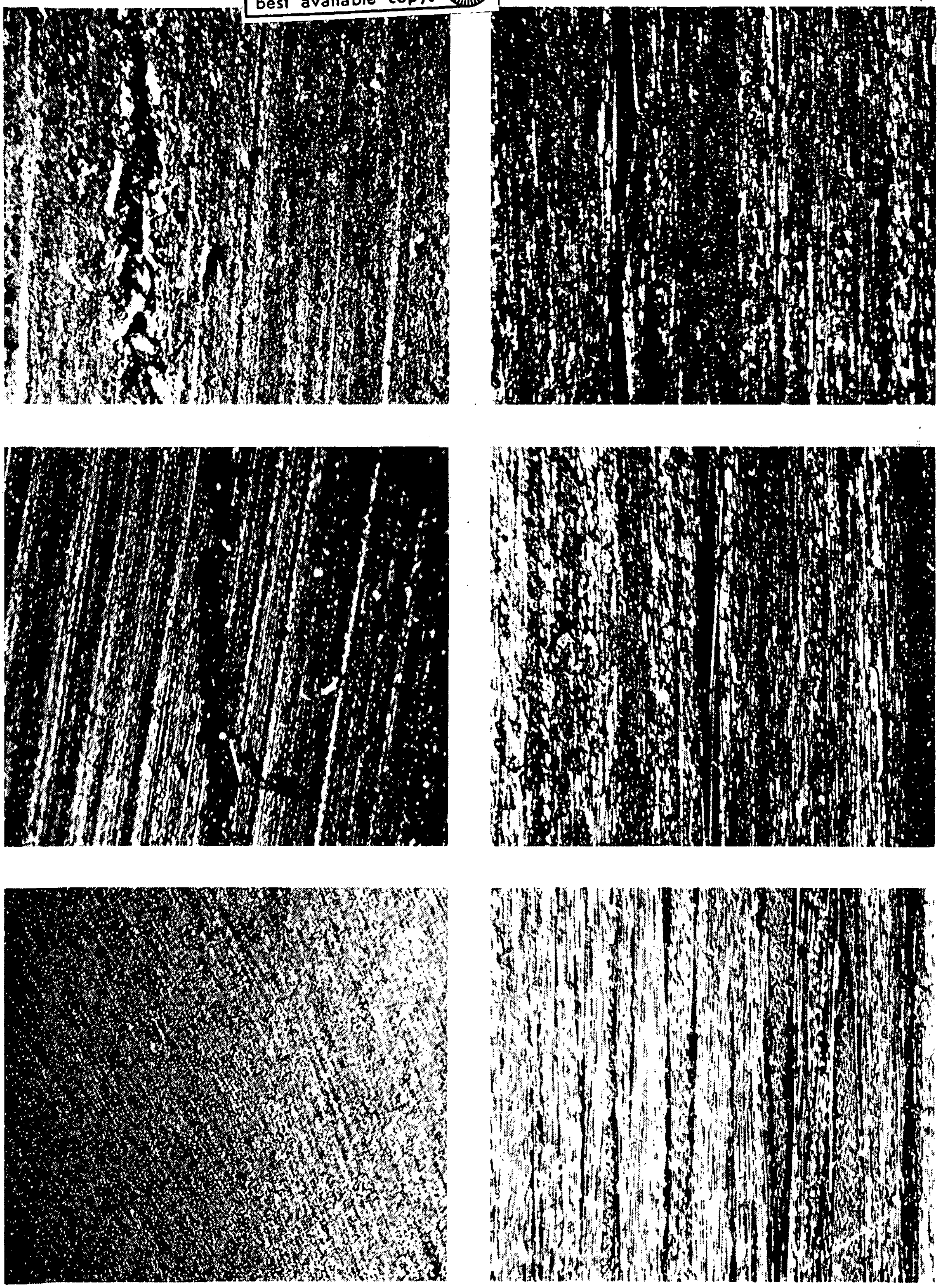
Prepreg Sheet or Basic Panel No.	Resin Flow, %(2)			Volatiles, %(3)			Resin Solids, %			Prepreg Wt gm/sq/in.			Fiber Wt gm/sq in.		
	P. O. Req	G. E.	NR/SD	P. O. Req	G. E.	NR/SD	P. O. Req	G. E.	NR/SD	P. O. Req	G. E.	NR/SD	P. O. Req	G. E.	NR/SD
PG19-1-22	20±5		11.53 20.90 16.75 16.39 Av	4.0	6.3 8.6 7.5 Av	4.73 4.46 5.76 4.98 Av	44±3	44.2	37.70 31.20 44.98 37.96 Av	Not Stipulated	0.1506	0.1434 0.2287 0.1111 0.1611 Av	Nominal 3.6 Tows per Incl(4)	0.0840	0.0609 0.1734 0.0564 0.0969 Av
PG 19-2-22	20±5	17.6	17.29 22.00 9.84 16.38 Av	6.0	9.9 8.2 9.0 Av	5.42 13.40 5.84 8.22 Av	44±3	43.0	38.19 37.60 27.40 34.39 Av		0.1444	0.1203 0.2244 0.1544 0.1664 Av		0.0826	0.0525 0.1148 0.0513 0.0729 Av
PG 19-3-22	20±5	18.7	13.49 16.72 12.83 14.35 Av	8.0	9.5 10.0 9.8 Av	1.65 13.28 5.00 6.64 Av	44±3	39.3	36.35 40.62 40.87 39.28 Av		0.1465	0.1356 0.1465 0.1254 0.1482 Av		0.0888	0.0515 0.0489 0.0575 0.0526 Av
PG 19-4-22	20±5	21.7	21.73 18.60 12.90 17.74 Av	10.0	9.6 10.5 10.5 Av	6.01 7.66 4.90 6.19 Av	44±3	43.0	44.12 45.23 40.98 43.44 Av		0.1465	0.1809 0.1761 0.1580 0.1717 Av		0.0819	0.0901 0.0932 0.0723 0.0852 Av

(1) Batch HTB - 100B 170069
(2) Determined at 100 psi, 350 F
(3) Determined at 600 F for 15 minutes
(4) Corresponds to 0.0855 gm/sq in.

Table 3-6. Short Beam Shear Strength Results, Gemon L/HTS

Panel Design / Property		PG 19-1-22 A	PG 19-1-22 B	PG 19-1-22 C	PG 19-2-22 A	PG 19-2-22 B	PG 19-2-22 C	PG 19-3-22 A	PG 19-3-22 B	PG 19-3-22 C	PG 19-4-22 A	PG 19-4-22 B	PG 19-4-22 C
Max P.C. temp, °F		500	565	600	500	565	600	500	565	600	500	565	600
Density, lb/cu in.		0.0552	0.0547	0.0522	0.0558	0.0553	0.0514	0.0559	0.0566	0.0498	0.0557	0.0559	0.0539
Resin content, w/o		39.4	38.1	39.8	34.4	37.0	30.2	34.5	32.1	32.2	35.6	36.6	37.2
Fiber content, v/o		52.9	54.9	55.0	58.5	55.7	66.4	58.3	60.7	65,4	57.1	55.7	56.6
Void content, v/o		0.8	2.1	4.4	1.4	6.0	0.5	1.2	0.8	12.7	1.2	0.5	3.9
Wt loss in P.C., %		1.36	2.84	5.25	1.20	2.79	5.68	1.30	2.56	5.14	1.10	2.53	4.12
Transverse shrinkage in P.C., %		0.87	1.19	0.80	0.68	1.14	0.57	0.94	1.33	0.71	0.86	1.23	0.61
Ply thickness, mil		5.7	5.9	6.0	6.0	5.7	5.8	5.5	4.9	5.8	5.8	5.4	5.8
Short Beam Shear Strength, ksi	RT	14.1 12.2 12.8	15.0 13.3 12.6	7.5 10.8 5.9	10.1 11.9 12.2	13.0 13.5 14.1	5.9 7.3 6.6	13.3 13.1 13.5	15.8 15.3 18.0	6.3 5.2 6.0	14.6 12.9 12.5	9.3 11.7 12.9	12.4 10.4 10.9
	Av	13.0	13.6	8.1	11.4	13.5	6.6	13.3	16.4	5.8	13.3	11.3	11.2
	500 F	8.4 7.1 8.3			6.8 7.6 7.2			6.9 6.2 6.5			7.7 7.2 7.9		
	Av	7.9			7.2			6.5			7.6		

MAX POST-CURE TEMP 500 F 565 F 600 F



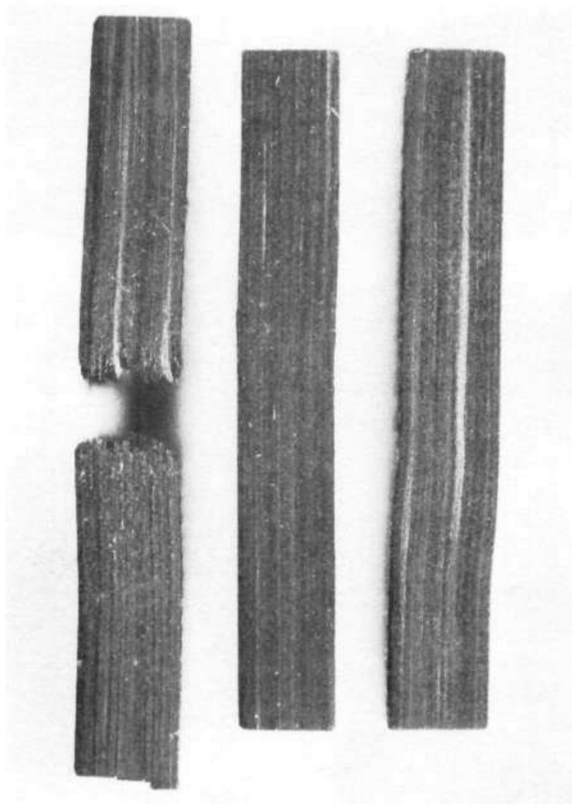
END VIEW

EDGE VIEW

Figure 3-4. Effect of Post-Cure Temperature on Splitting Tendencies, Gemon L/Graphite Composite (40X Photos)

TEST TEMPERATURE

RT	RT	500 F



TENSILE & SHEAR	SHEAR ONLY	SHEAR ONLY

Figure 3-5. Failure Modes, 500 F Post-Cured Gemon L/Graphite
Short Beam Shear Specimens

The RT and 500 F short beam shear strength results on the Gemon L system, freedom from microcracks, and low void content of the low pressure laminated panels, confirmed Gemon L as the resin selection for this temperature range.

Thermal Cycling Effects

Crossplied laminate panels four inches square and 0.080 inches thick were prepared for these studies by the following processes:

Modmor II/Pyralin 4707:	Process 2
Modmor II/P13N:	Process 10
Modmor II & HTS/Gemon L:	Process 15, using a perforated aluminum caul plate and a maximum post-cure temperature of 500 F

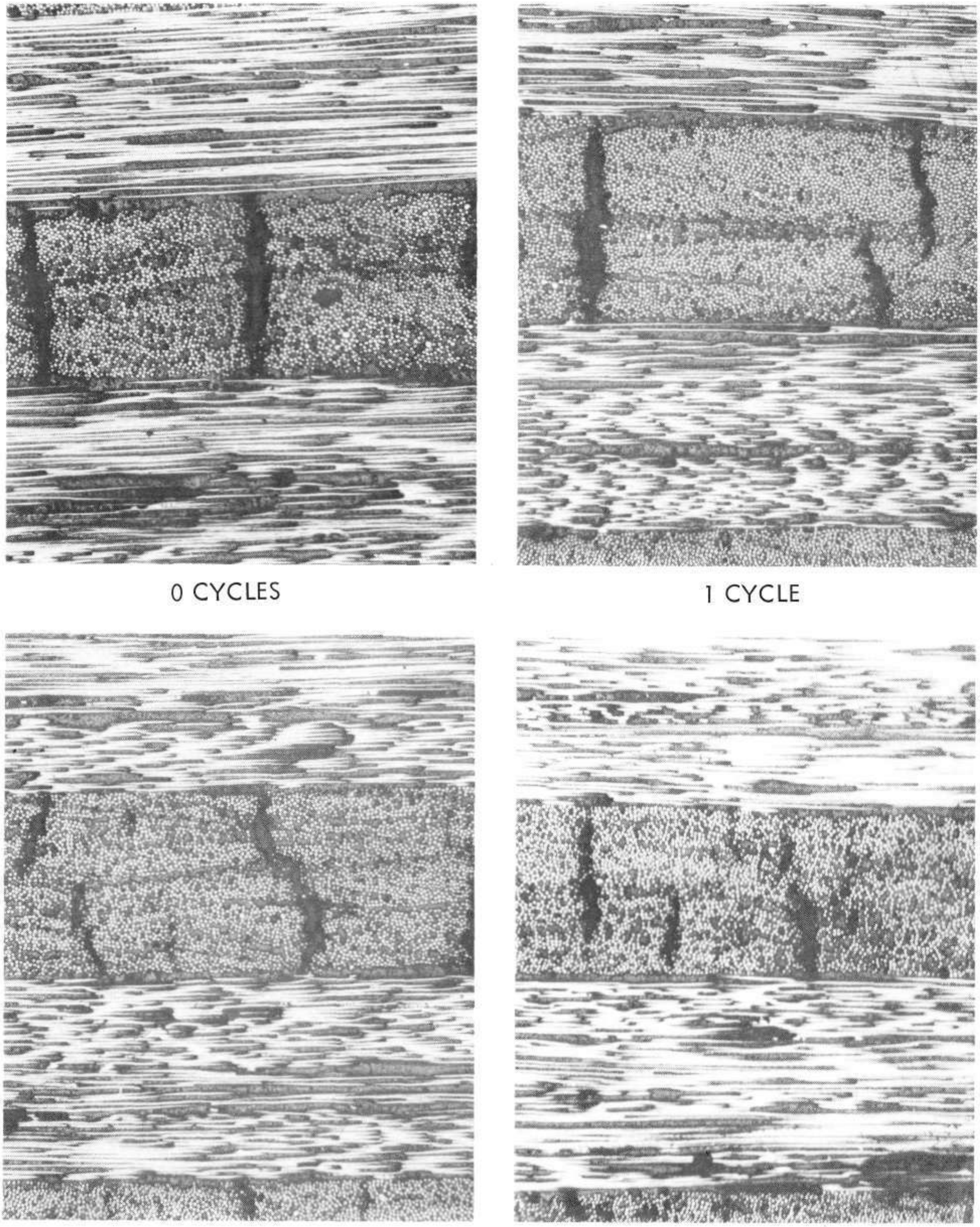
One specimen of each of these material configurations was subjected to thermal cycling and samples taken for visual inspection after 1, 5, and 10 cycles. The Gemon L specimens were cycled between -65 and 500 F and the Pyralin 4707 and P13N ones between -65 and 600 F. No evidence of degradation was noted in any of the specimens after 1, 5, and 10 cycle exposures as evidenced by the photomigrographs of Figures 3-6 through 3-9. The system eventually chosen for fabrication of the wing box beam, Gemon L/ Modmor II, shows the highest incidence of micro-voids. However, the measured voids do not exceed the permissible percentage and show no apparent increase in number or size because of thermal cycling.

The lack of thermal cycling to produce adverse effects on composite mechanical properties was also displayed by the results of short beam shear strength tests performed on unidirectional laminates, Table 3-7.

Fiber Selection

The intended test matrix for this task, Table 3-8, reflects the a priori design decision to limit the graphite fiber choice to two "high strength" materials. It further reflects the plan to use mechanical properties data generated in this study as the basis for material specification and design values for reasons of economy. The flexural, transverse, and ±45° properties, therefore, are established only on the chosen reinforcing fiber system, be it Modmor II or HTS. Due to the questionable nature of rail shear type results, it was planned to determine in-plane shear properties from tensile results and appropriate transformation equations.

Continuous filament prepreg of HTS and Modmor II/Gemon L was ordered from General Electric to stipulated requirements and three pounds of material for each system were received on November 11, 1970.



0 CYCLES

1 CYCLE

5 CYCLES

10 CYCLES

Figure 3-6. Effect of Thermal Cycling from -65 to 500 F on Microstructure of Cross-Plied Composite, 4707/Modmor II, Panel PD 32-1

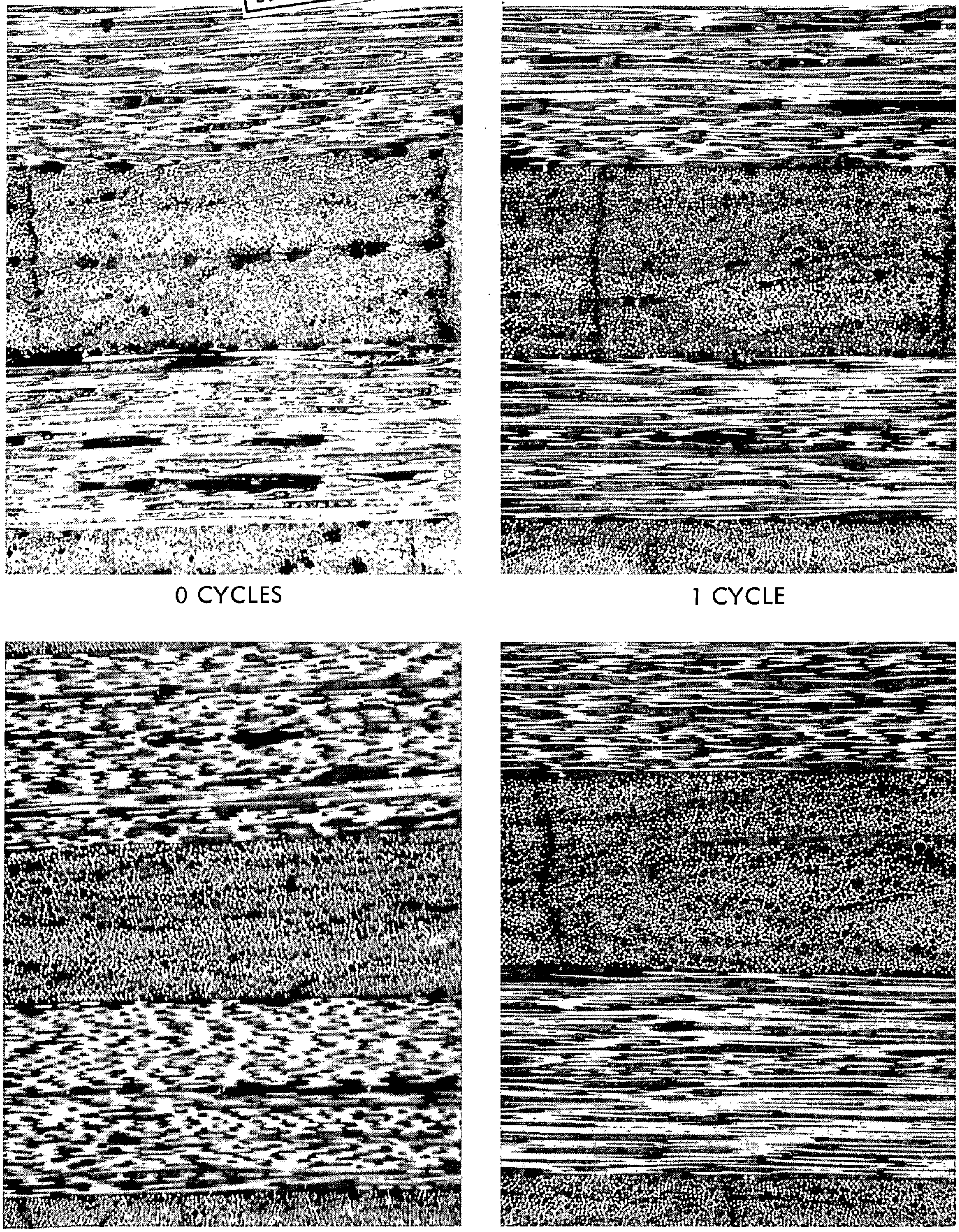
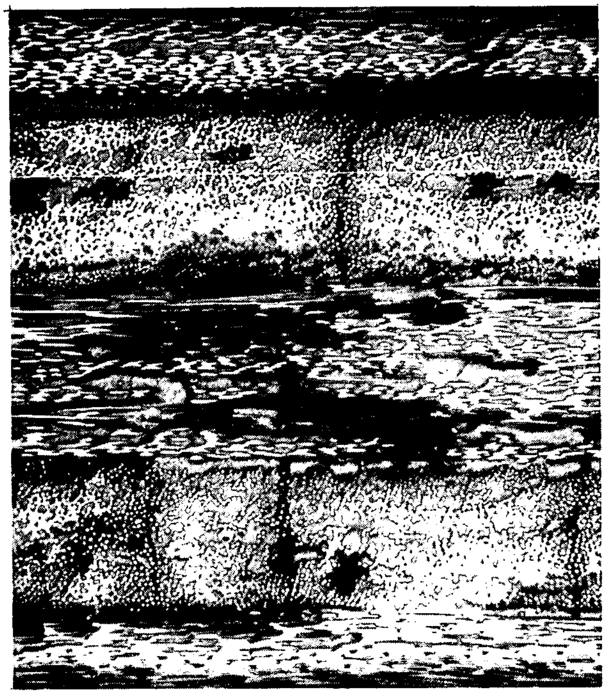
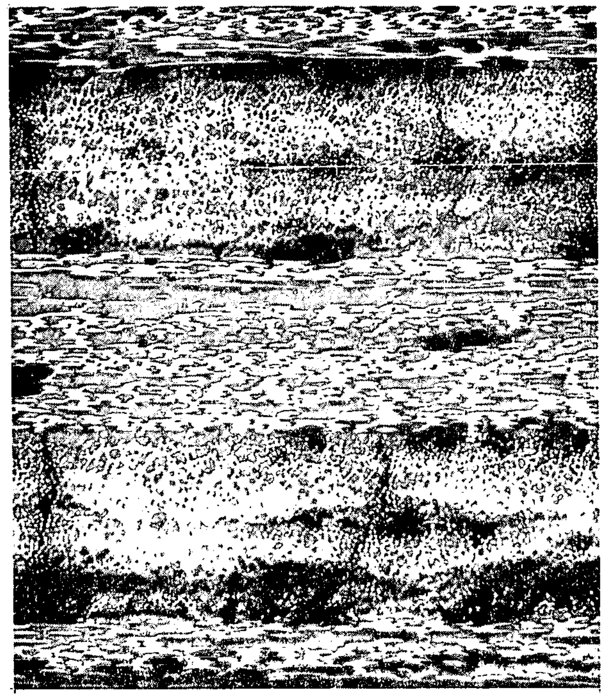


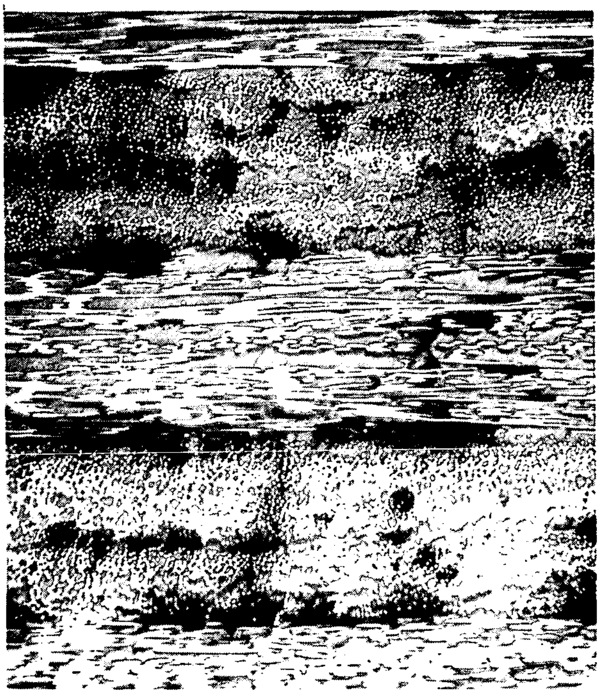
Figure 3-7. Effect of Thermal Cycling from -65 to 500 F on Microstructure of Cross-Plied Composite, P13N/Modmor II, Panel P 13



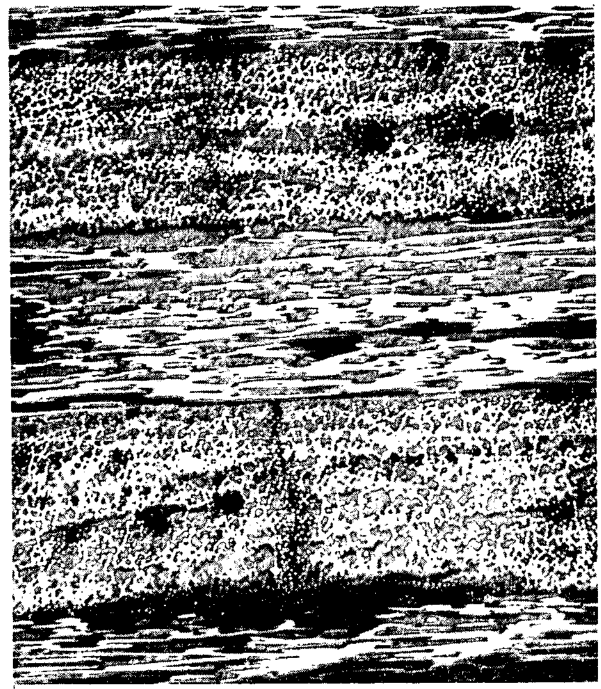
0 CYCLES



1 CYCLE



5 CYCLES



10 CYCLES

Figure 3-8. Effect of Thermal Cycling from -65 to 500 F on Microstructure of Cross-Plied Composite, Gemon L/Modmor II, Panel PG 21-4-29A

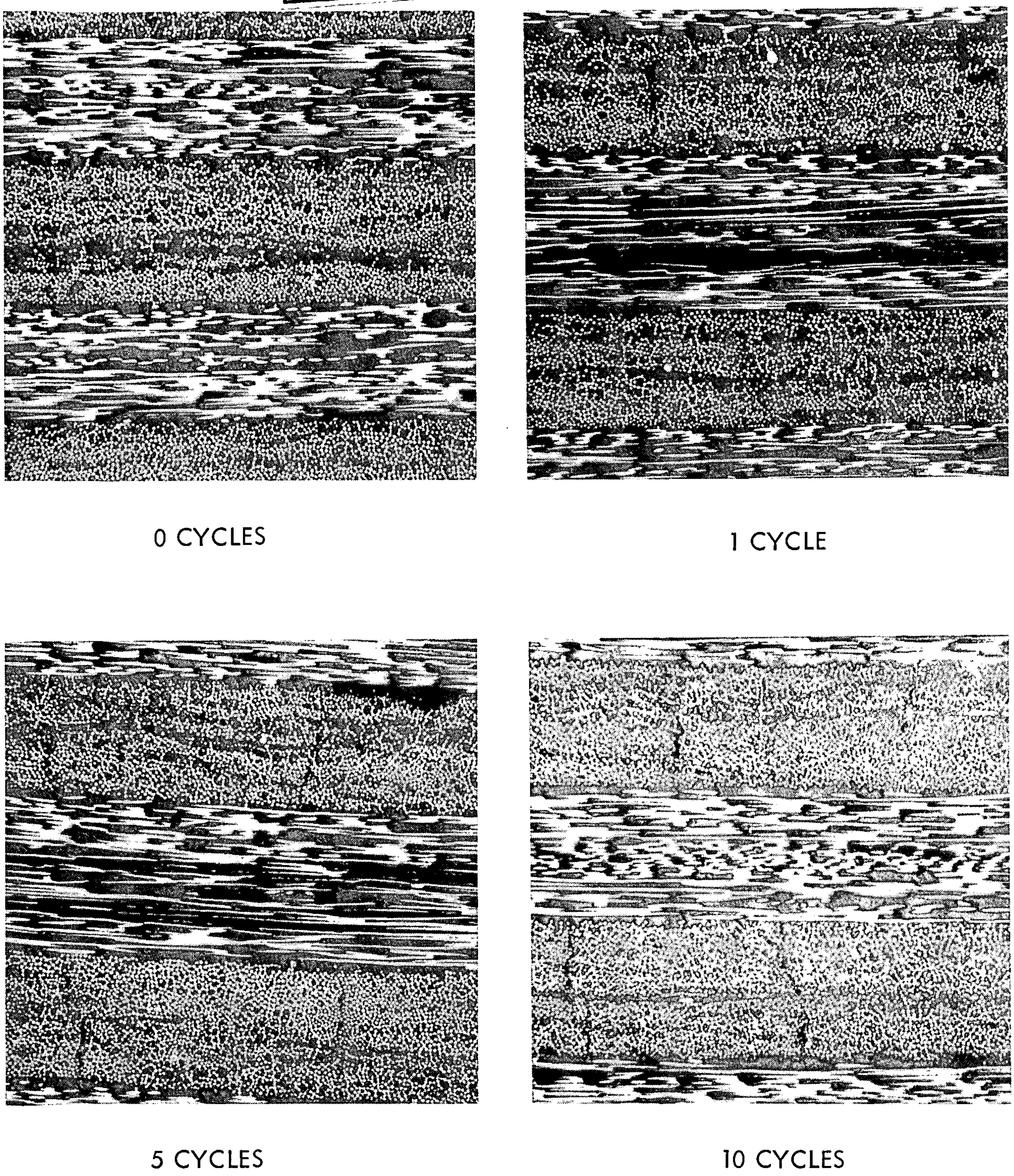


Figure 3-9. Effect of Thermal Cycling from -65 to 500 F on Microstructure of Cross-Plied Composite, Gemon L/HTS, Panel PG 23-1

Table 3-7. Temperature Cycling Effects, Short Beam Shear Strength

Panel Designation and Resin Type	Short Beam Shear Strength After Cycling, Ksi	
	RT	500 F
PD 26-1-6(1) 4707	7.62 7.79 7.70 ——— 7.70 Av	
PG 15-2-22(1) Gemon L	11.8 11.5 13.1 ——— 12.1 Av	5.05 5.06 4.76 ——— 4.96 Av
P 12(1) P13N	11.5 12.1 11.7 ——— 11.8 Av	
PG 19-2-22A(2) Gemon L	11.0 10.7 11.2 ——— 11.0 Av	7.2 7.3 6.7 ——— 7.1 Av

(1)After ten cycles from -65 F to 565 F
(2)After ten cycles from -65 F to 500 F

The results of receiving inspection tests at NR/SD and those of the General Electric Certificates of Test are presented in Table 3-9. Large differences are apparent, although sampling and testing procedures are essentially identical with one exception. The exception is the method used for resin extraction (NR/SD used nitric acid and G. E. used the newer and faster sulphuric acid/hydrogen peroxide method). The nitric acid digestion procedure was suspected to be more severe in terms of attack on the fiber. NR/SD tests on raw Modmor II and HTS fibers resulted in weight losses of 2 percent and 0.13 percent respectively, not significant in terms of receiving inspection tolerances.

Table 3-8. Intended Test Matrix, Fiber Selection

Test Type	Fiber Orientation	Fiber Type	Test Temp	Specimen: No.	Specimen: Dimensional Characteristics	Specimen: Type	Specimen: Tabs Req	Instrumentation: None Req	Instrumentation: Deflectometer	Instrumentation: S/G	Instrumentation: Extenso-Meter
Basic											
Tensile strength	0°	Mod II & HTS	RT & 500 F	3 ea	7-1/4" x 1/4" x 8 plies[1]	Straight sided	X	X			
Compressive str and mod	0°				3" x 1/2" x 8 plies[1]	Finger supported					X
SBS	0°				0.62" x 1/4" x 13 plies[2]			X			
Replica											
Tensile strength	0°				7-1/4" x 1/4" x 8 plies[1]	Notched	X	X			
Compressive strength	0°				3" x 1/2" x 8 plies[1]	Finger supported		X			
SBS	0°				0.62" x 1/4" x 13 plies[2]			X			
Additional											
Flex str and mod	0°				4" x 1/2" x 13 plies[2]				X		
Tens str mod and Poisson's ratio	0°	Mod II or HTS		2 ea	7-1/4" x 1" x 8 plies[1]	Straight sided	X			X	X
Tens str and mod	90°			2 ea	7-1/4" x 1" x 8 plies[2]		X				X
Tens str and mod	±45°			3 ea	7-1/4" x 1" x 8 plies[1]		X				X
Comp strength	0°			2 ea	2-1/2" x 1/2" x 13 plies	Part released (COL)	X	X			
Comp str and mod	90°			2 ea	3" x 1" x 8 plies	Finger supported					

Nominal Thickness
[1] 0.042 in.
[2] 0.068 in.

Table 3-9. Prepreg Properties, P.O. No. MIE-3XAV-635069[1]

Property	Volatiles, Wt %		Resin Solids, Wt %		Fiber Wt, gm/sq ft		Flow, Wt %	
P.O. Req't	6 to 10		44 ± 4		13.1 ± 0.65		20 ± 5	
Testing Activity	G.E.	NR/SD	G.E.	NR/SD	G.E.	NR/SD	G.E.	NR/SD
Material Tested								
Sheet 103070-D HTS Lot 2CT9A/56R Resin Lot/Batch 70069	7.1 8.0 7.5	5.88 12.49 11.15 9.84	41.0 42.0 41.5	34.59 34.84 41.05 36.56	13.1 12.7 12.9	8.4690 12.2256 12.1248 10.9488 11.7187	24.9	12.13 23.65 5.18 13.65
Sheet 103070-E As above	8.8 10.0 9.4	41.4 45.3 43.4	42.2 47.4 44.8		12.7 12.7 12.7		24.2	
Sheet 103070-F As above	6.1 7.3 6.7		41.4 45.3 43.4		12.7 12.5 12.6		22.9	
Sheet 110270-A As above	8.5 8.4 8.4		40.8 42.4 41.6		12.5 12.7 12.6		15.3	
Sheet 110270-C HTS Lot 2CT9A/22AR Resin Lot/Batch 70069	8.9 10.3 9.6	11.14 5.13 8.12	41.2 40.4 40.8	32.84 36.89 34.86	13.4 12.9 13.2	9.9648 15.5376 12.7512	18.8	13.76 15.10 25.10 17.98
Sheet 110270-D As above	9.9 9.7 9.8		40.4 40.7 40.6		12.7 12.7 12.7		17.7	
Sheet 110270-E HTS Lot 2CT9A/22AR Resin Lot/Batch 70069	9.6 10.0 9.8		40.7 41.8 41.3		12.6 12.4 12.5		15.8	
Sheet 110270-F	10.0 9.8 9.9		43.1 43.2 43.2		13.1 12.8 13.0		17.4	
Sheet 110470-A Modmor II Lot 0235/0539/522 Resin Lot/Batch 70069	10.1 8.9 9.5	5.35 11.12 5.12 7.19	44.3 40.1 42.2	39.98 41.96 36.32 39.42	12.9 12.4 12.7	9.1296 14.6016 9.1728 10.9680	15.3	7.05 19.29 15.23 13.85
Sheet 110470-B As above	9.4 10.6 10.0		42.8 44.9 43.9		12.8 12.2 12.5		19.7	
Sheet 110470-C Modmor II Lot 0189/C35/S9 Resin Lot/Batch 70069	7.4 6.4 6.9		43.4 40.1 41.8		12.8 13.8 13.3		15.6	
Sheet 110470-D As above	8.9 7.9 8.4		41.5 40.0 40.7		12.8 12.6 12.7		25.0	
Sheet 110470-E Modmor II Lot 0193/C37/S20 Resin Lot/Batch 70069	6.1 6.6 6.4	5.20 8.36 5.89 6.25	40.9 42.2 41.6	39.11 36.48 38.69 38.09	12.8 12.1 12.5	10.5120 12.1536 11.3760 11.3472 12.9164	22.4	23.45 14.31 10.54 16.10
Sheet 110470-F As above	7.9 7.1 7.5		43.6 43.9 43.7		12.8 12.2 12.5		23.8	
Sheet 110570-A As above	6.7 6.9 6.8		39.9 41.6 40.8		13.5 13.1 13.3		18.9	
Sheet 11057-B As above	9.8 10.2 10.0		44.9 44.6 44.7		13.4 13.0 13.2		19.5	

(1) Underlined figures are averages of first determinations at several locations. Figures below averages, if appearing, are second determinations at a specific location.

All panels for the fiber selection test program were fabricated per Process 15 (Appendix A) with a 500 F termination temperature and a perforated aluminum caul plate. The panels of unidirectional HTS/Gemon L laminate, both 8 and 13 ply, blistered while all Modmor II/Gemon L panels came through in excellent condition. Investigation of the possible causes for blistering of the HTS/Gemon L panel revealed that the only traceable difference between this and the material previously used in the program was a slight modification in the fiber surface treatment by Courtauld, the fiber manufacturer. Insufficient fiber washing, since corrected, has rather definitely been identified as the problem source, in similar observations by J. Hertz, Reference 5.

In view of the problem associated with HTS/Gemon L and the time and effort which would be involved in requalifying the material, it was decided that no further work would be expended on this system.

Physical properties of the panels produced in direct support of the combined fiber selection/basic mechanical properties tasks reported in this section are presented in Table 3-10. In the isolated cases (short beam shear and flexural strength) where already existing data were used as basic set values, panel descriptions may be found in Table 3-5.

Results of the 0° tensile, compressive, and short beam shear basic and replica determinations are shown in Table 3-11. Tensile elastic modulus determinations, not planned for this series, but performed are also shown. All of the data are in reasonable agreement with analogous values of high strength graphite fiber/epoxy composites, the 500 F properties of the Gemon L system comparing with 350 F values of epoxy ones.

Difficulties associated with testing of the 0° tensile and compressive specimens were resolved as follows:

Tensile Testing

Initial difficulties in elevated temperature tensile testing (500 F and above) of graphite/PI composites were solved by use of fabric/PI end tabs and by installation of 10,000-pound capacity spring loaded grips, Templin, Heavy Duty, Model TG-10535, within the test chamber. The grips employ 2.5-inch long, 1-inch wide serrated facings. The tabs were bonded to the specimens with FM-34 adhesive.

Compression Testing

Due to difficulties in compression testing of advanced composite materials no standardized test methods have as yet been developed. In particular, the load intensities required for edgewise testing of 0-degree oriented laminate coupons often result in failure modes other than the one desired.

Table 3-10. Gemon L/Modmor II Panel Characteristics

	Panel No.	Ply		Density lb/cu in.	Fiber[1] Content V/O	Calculated[1] Void Content V/O	Ply Thickness 0.001 in.	Heat Rise in Cure °F/min.	Remarks
		No.	Array						
Set 1	PG 21-2-29	13	0	0.0555	51.4	-2.0	6.0	7.24	
	PG 21-4-29	8	±45	0.0555	47.6	-2.8	5.8	7.24	
	PG 23-2-29A	8	0/90	0.0555	56.2	-1.0	5.3	8.4	
	PG 27-1-22	8	0	0.0556	56.1	-0.8	5.7	6.56	
	PG 28-1-22	8	0	0.0552	59.5	0.7	5.4	5.21	
	PG 29-1-22	8	0	0.0556	61.0	0.5	5.5	5.5	
	PG 29-2-22	13	0	0.0552	56.0	1.4	6.3	5.5	
Set 2	PG 31-1-22	13	0	0.0536	52.6	1.6	6.5	2.05	
	PG 31-2-22	13	0	0.0524	53.9	1.9	6.5	2.05	
	PG 33-1-19	13	0	0.0544	52.8	0.5	6.1	6.0	
	PG 36-1-22	13	0	0.0545	57.6	1.4	5.8	5.8	Short P.C.
	PG 36-2-22	13	0	0.0547	58.4	1.2	6.1	5.8	Short P.C.
	PG 41-1-19	8	0	0.0546	59.6	1.8	5.1	6.3	
	PG 41-19-1	8	0	0.0546	58.1	3.3	5.1	6.3	

(1)Calculated on the basis of fiber density $\rho = 0.0617$ lb/cu in.

Table 3-11. Tensile, Compressive, and Short Beam Shear Properties of Parallel Oriented Gemon L/Modmor II Composites, 0-Degree-Direction Fibers

	Basic Set		Replica Set	
Property	RT	500 F After 1/2 Hr at 500 F	RT	500 F After 1/2 Hr at 500 F
Tensile strength, ksi	169.5	151.1	172.1	169.3
	154.6	148.0	182.2	151.1
	156.1	152.7	156.3	142.6
	149.6	137.7		
	157.4*	147.3*	170.2*	154.3*
Modulus, msi	18.5			
	19.3			
	19.7			
	20.0			
	19.3*			
Compressive strength, ksi	135.9	--(1)	134.4	54.8
	118.8	57.5	141.8	59.4
	122.7	72.9	139.4	56.9
	125.8*	65.2*	138.5*	57.0*
Modulus, msi	16.0	--(1)	17.4	15.9
	15.7	19.2	--(2)	18.8
	16.3	17.4	17.3	17.8
	16.0*	18.3	17.3*	17.5*
Short beam shear strength, ksi(3)	12.6	8.1	10.98	7.00
	15.0	7.8	12.69	7.50
	13.2	7.2		6.85
	13.6*	7.7*	11.84*	7.12*

*Average
(1) Bad alignment; no test
(2) Extensometer slipped
(3) Basic Set: Panel PG-21-2-29; Replica Set: Panel PG-36-2-22

NR/SD has been using a modification of the lateral leaf spring support method specified in ARTC-11, Method 1. This modification provides a four-sided, potted end support for the specimen to forestall brooming of the ends, which results in premature failure. This method, while an improvement, still has not always resulted in clean failures in past work on epoxy matrix materials. For PI work it was decided to insure that all of the leaf springs provide their assigned share of the lateral support during test. It was determined that to achieve this the flatness of 0-degree specimen faces must be controlled by grinding to within 0.0005 inches. By exercising this flatness control compressive failure of the specimens was achieved within the gage length in all cases.

Additional Tests

Flexural properties determinations are reported in Table 3-12. Several panels were examined for longitudinal flexural strength to provide a reasonably broad data base for setting realistic minimum requirements in a material procurement specification and for in-process control coupons.

Additional tensile tests were determinations of strength, elastic modulus, and Poisson's ratio on 0-degree orientations in both the longitudinal and transverse directions and on ±45 degree orientations. The results are shown in Table 3-12. The Poisson's ratios were read at stress levels of 20 percent of ultimate. Figure 3-10 is a plot of Poisson's ratio obtained on typical specimens at various stress levels and indicates a strong dependence on the stress level. The stress-strain data for these specimens are shown in Figures 3-11 through 3-14.

Shear Modulus

Data analysis by various investigators has raised several questions concerning the accuracy of rail shear test data. The points in question involve shear lag phenomena, the introduction of the load into the specimen by the grips, and the effects of internal ply orientation.

Because of the questionable accuracy of this test procedure, it was decided to compute the shear modulus from the elastic constants obtained from tests of uniaxial and ±45-degree orientations.

Transformation relations based on laminated plate theory, when applied to the ±45-degree laminate, give a relationship between the Young's modulus for the ±45-degree laminate, E_{xx}, and the shear modulus, G_{12}, as:

$$G_{12} = \frac{2U_1 \, E_{xx}}{8U_1 - E_{xx}}$$

Table 3-12. Additional Tests, Flexural Properties Data, Unidirectional Gemon L/Modmor II Composites

	RT	500 F After 1/2 Hr at 500 F	Panel No. Special Notes
Longitudinal flex. str., ksi	209.9	171.6	PG 21-2-29
	198.6	179.8	
	194.6	150.0	
	201.1*	167.2*	
Mod, msi	14.7	15.2	
	14.1	15.2	
	15.4	14.9	
	14.7*	15.1*	
Str, ksi	238	116	PG 39-3-22
	257	134	Meter Length
			Modmor II
	248*	125*	Prepreg of Low
			Resin
Mod, msi	18.40	15.59	Content (32 w/o)
	19.09	17.00	
	18.75*	16.30*	
Str, ksi			
	189	139	PG 42-3-19
	211	164	28 Hour Post-Cure
	195	136	
	198*	146*	
Mod, msi	18.33	18.08	
	19.75	19.02	
	18.74	17.60	
	18.94*	18.23*	
Transverse flex. str., ksi	9.61	3.45	PG 41-2-19
	7.25	3.09	
	8.43*	3.78*	
Mod, msi	0.46	0.120	
	0.29	0.081	
	0.37*	0.138	
		0.113*	

*Average

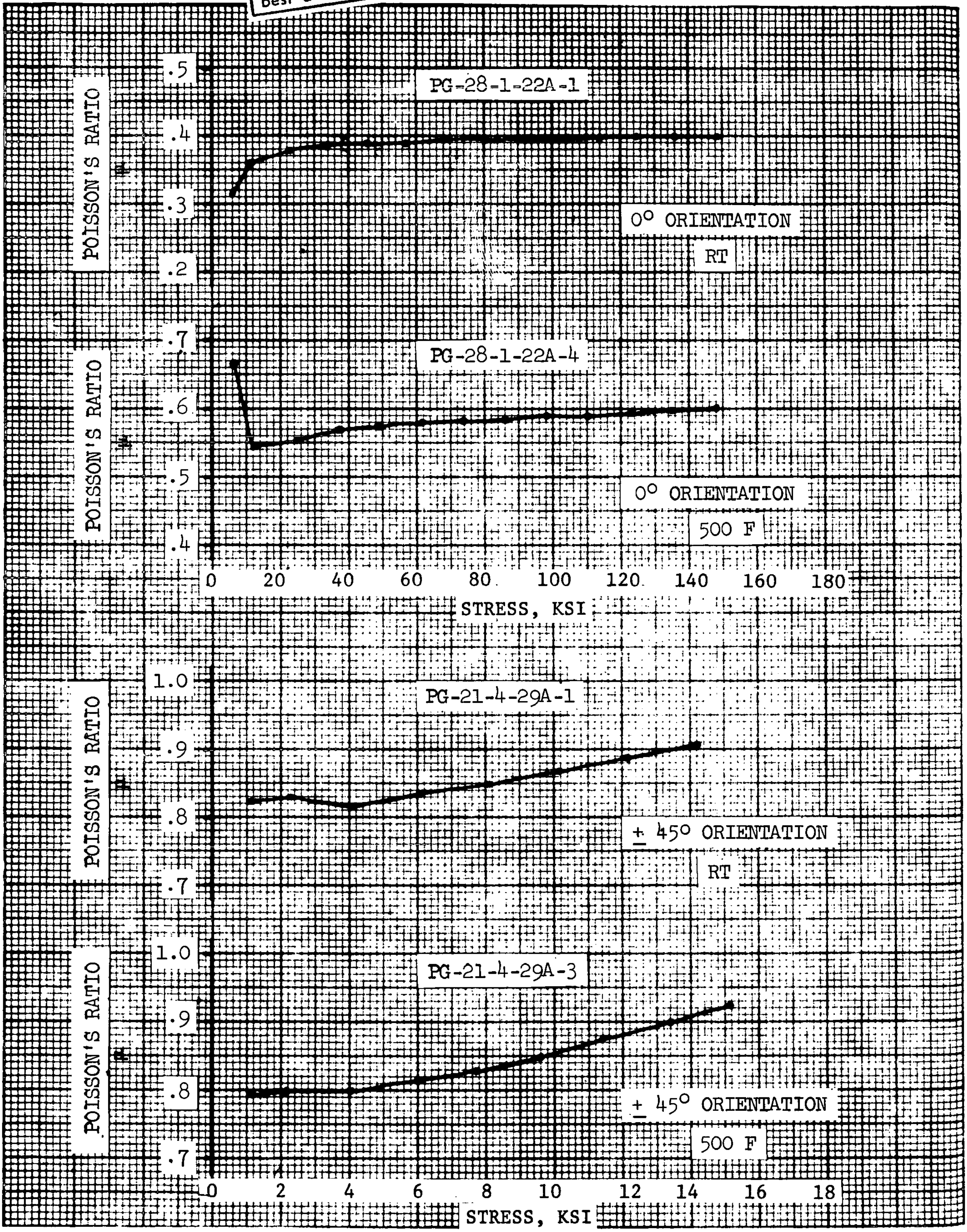


Figure 3-10. Poisson's Ratio, Gemon L/Modmor II Composite

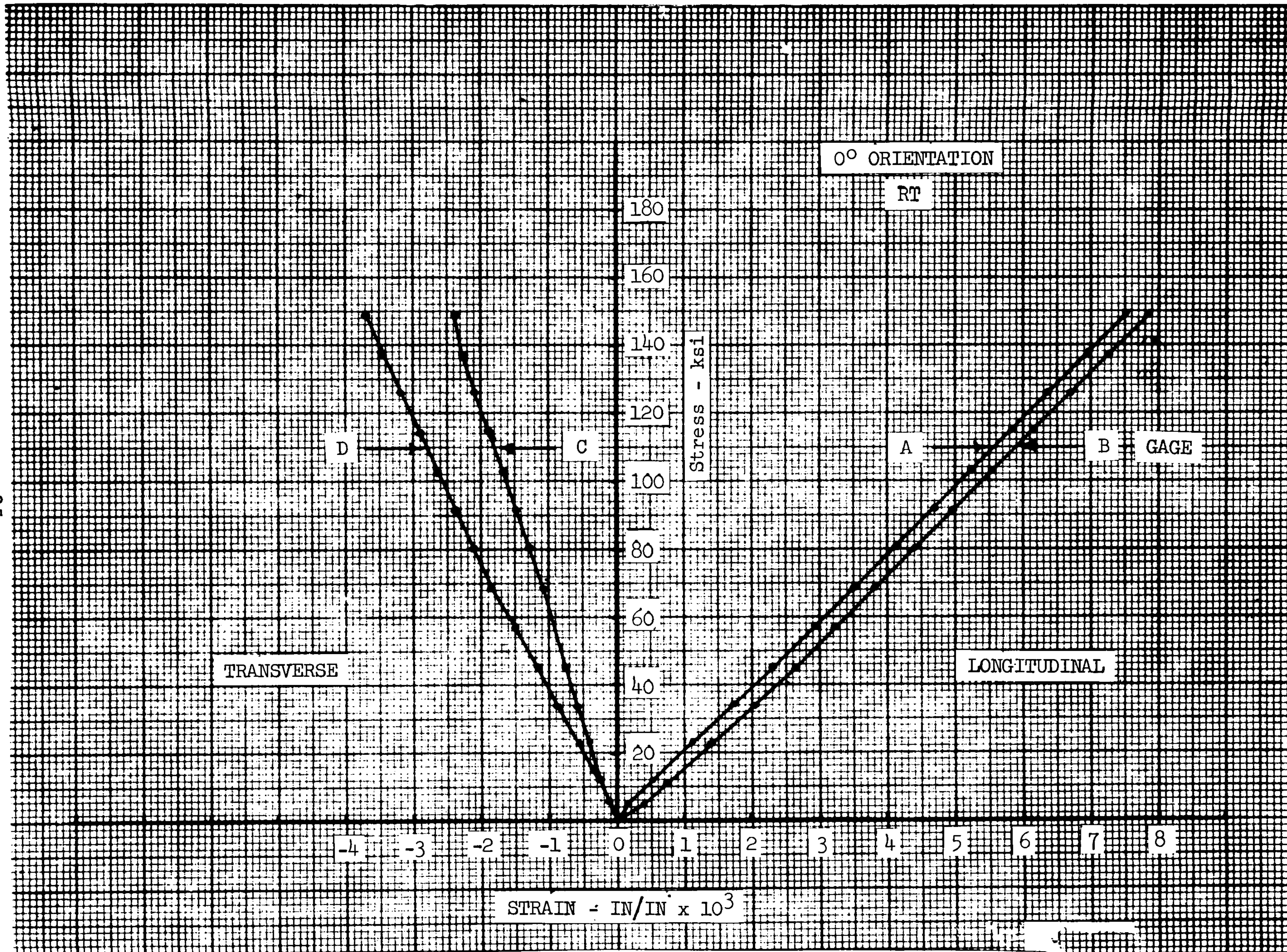


Figure 3-11. Stress Versus Strain, Gemon L/Modmor II Composite—0-Degree Orientation, Room Temperature

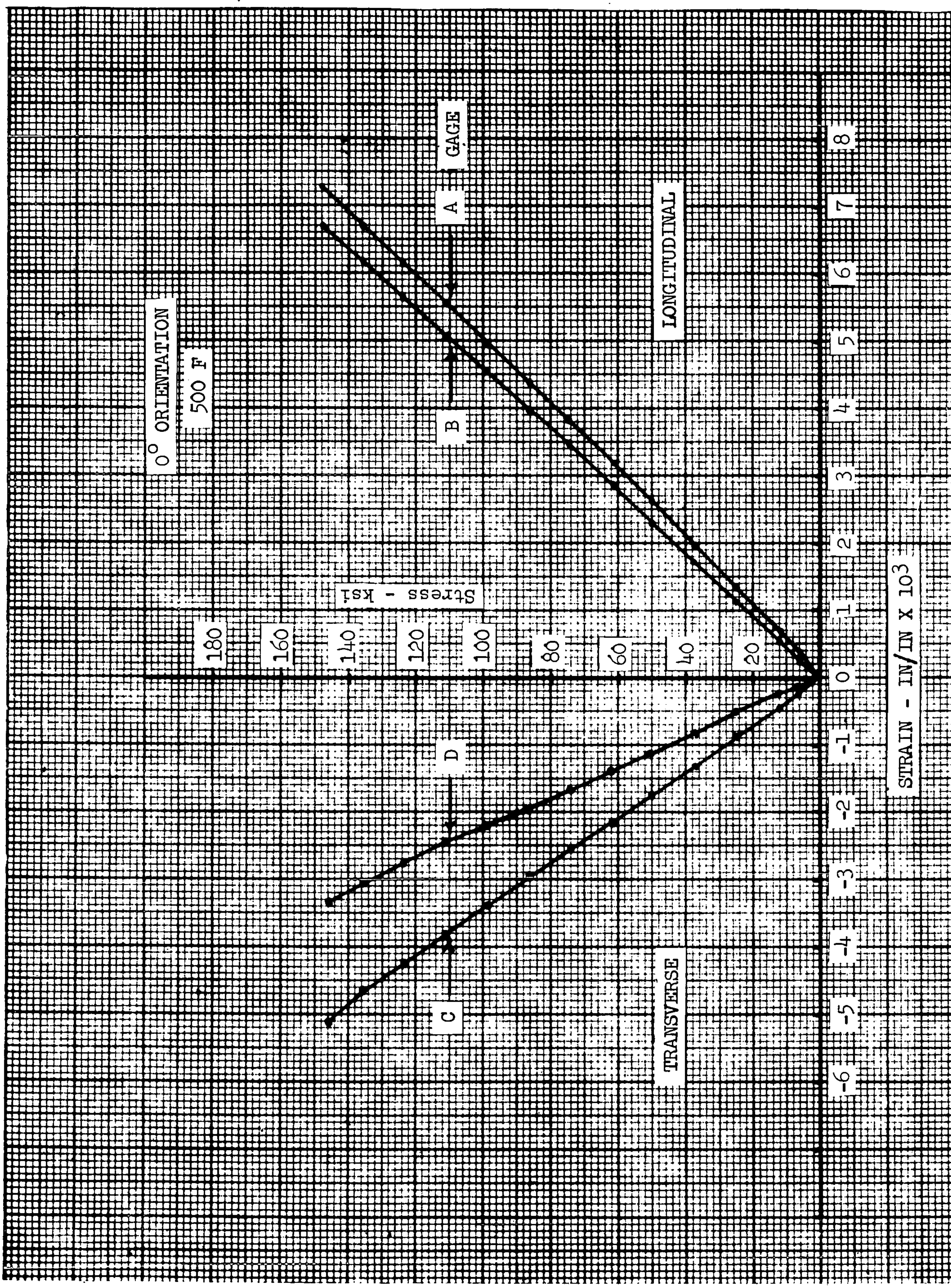


Figure 3-12. Stress Versus Strain, Gemon L/Modmor II Composite — 0-Degree Orientation, 500 F

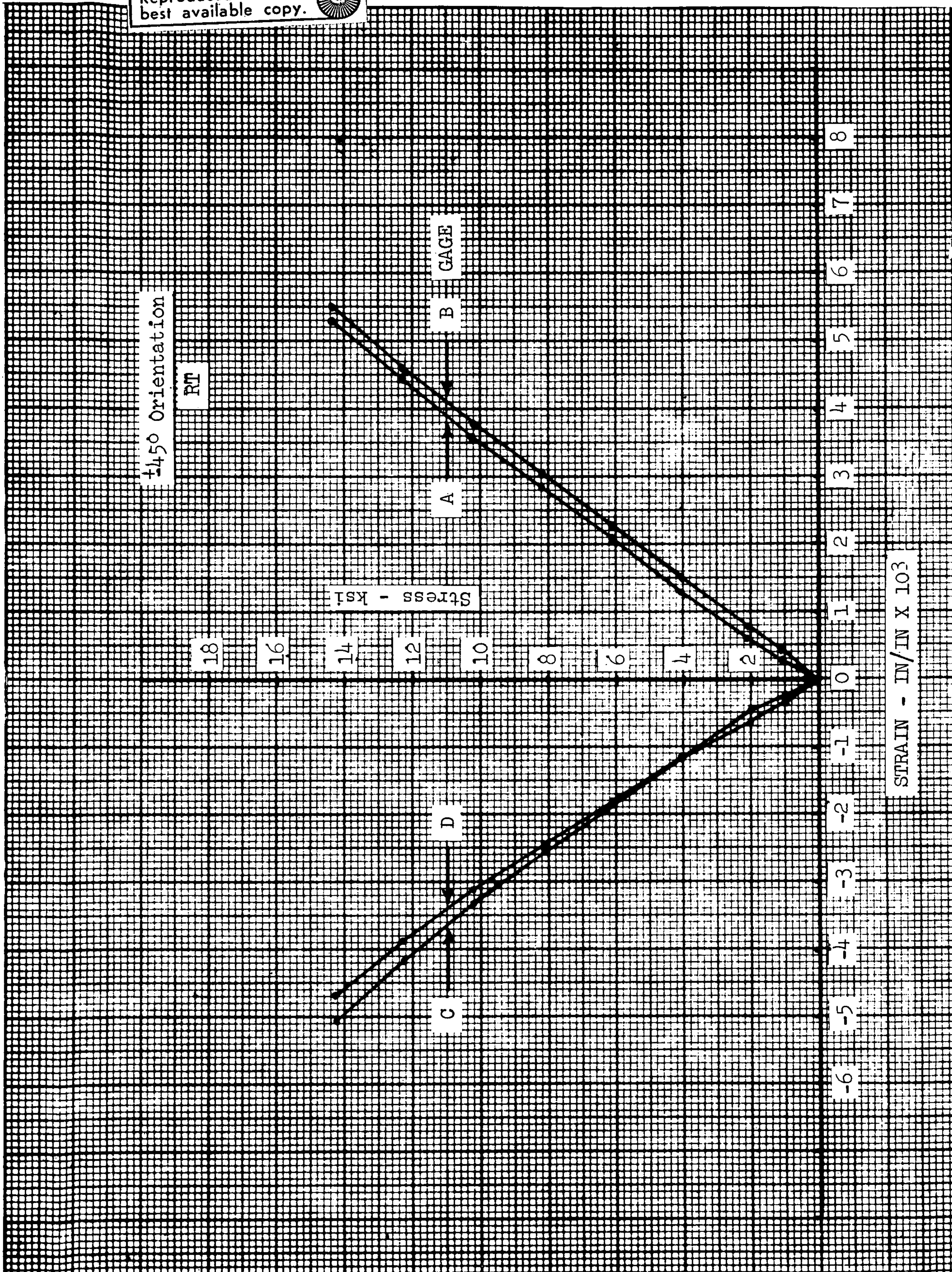


Figure 3-13. Stress Versus Strain, Gemon L/Modmor II Composite—±45-Degree Orientation, Room Temperature

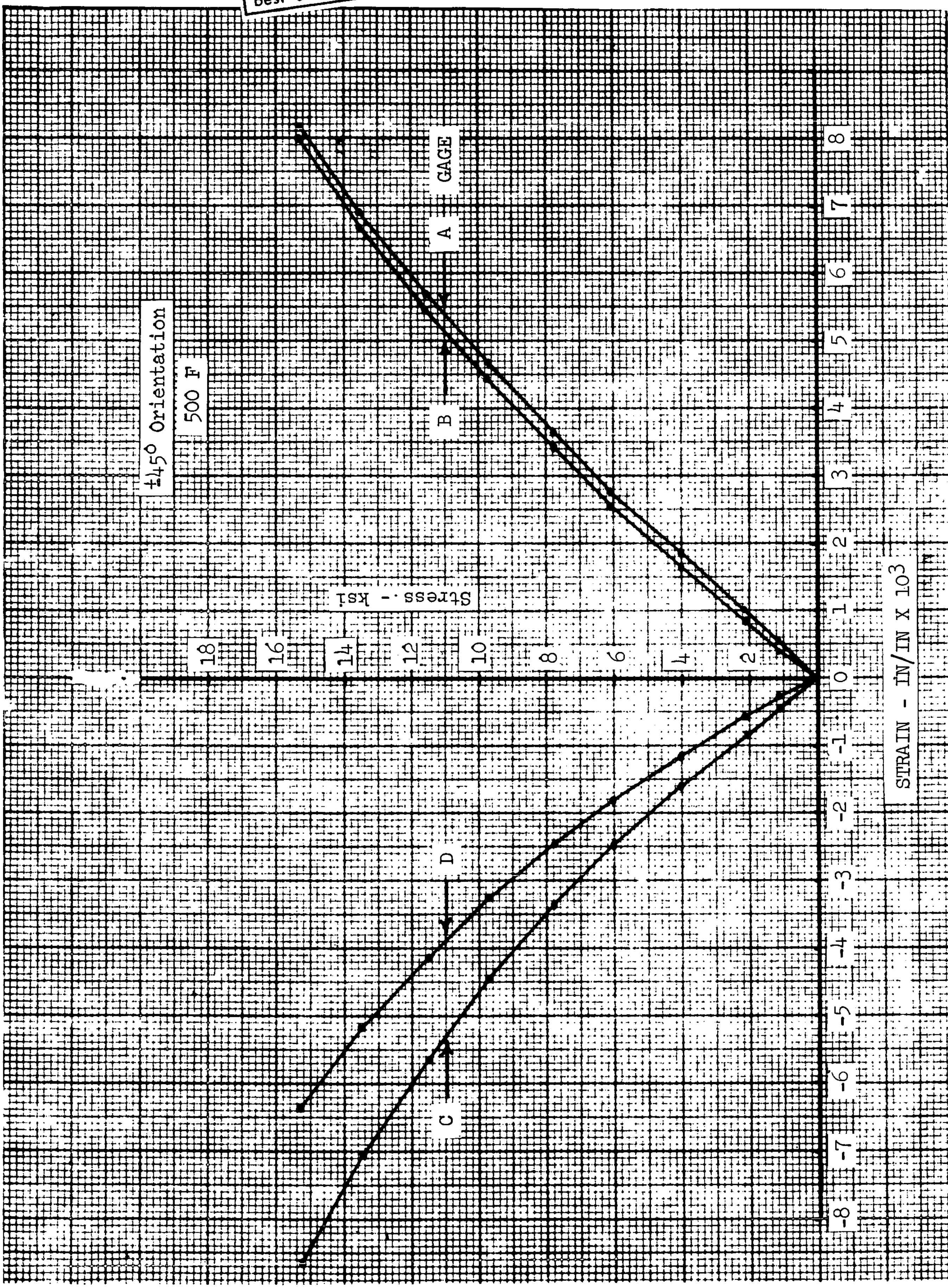


Figure 3-14. Stress Versus Strain, Gemon L/Modmor II Composite — ±45-Degree Orientation, 500 F

Table 3-13. Additional Tensile Properties, Gemon L/Modmor II Composites

Panel No.	Array Degrees	Tensile Strength Ksi RT	Tensile Strength Ksi 500 F	Tensile Modulus Msi RT	Tensile Modulus Msi 500 F	Poisson's Ratio RT	Poisson's Ratio 500 F
PG 28-1-22	0	See Table 3-11, Basic Set		18.5	19.0	0.36	0.46
				19.3	20.1	0.38	0.57
				19.7			
				20.0			
				19.3*	19.5*	0.37*	0.51*
PG 28-1-22	90	2.46	0.98	1.26		Not scheduled	
		2.39	0.94	1.43			
		2.34		1.29			
		2.39*	0.96*	1.32*	0.6*		
PG 21-4-29	±45	18.2	17.6	2.76	2.3	0.82	0.80
		17.7	16.9	3.22	2.25	0.79	0.73
		17.9*	17.2*	2.96*	2.3*	0.80*	0.76*

*Average

and

$$U_1 = \frac{1}{8(1 - \nu_{12}\,\nu_{21})}\left[E_{11} + E_{22} + 2\,\nu_{12}\,E_{22}\right]$$

Where:

E_{11} = unidirectional modulus measured in a longitudinal tension or compression test

E_{22} = unidirectional modulus measured in a transverse tension or compression test

ν_{12} = major Poisson's ratio

ν_{21} = minor Poisson's ratio = $\nu_{12}\,E_{22}/E_{11}$

E_{xx} = modulus in a ±45° tension or compression test

Using the average elastic constants of Table 3-12 and a value of 1.0 msi for E_{22} at 500 F (not experimentally determined), the following typical shear modulus values were computed:

	RT	500 F
G_{12}, ksi	0.81	0.64

Additional compressive data obtained are shown in Table 3-14. The "Columbus" specimen configuration (see Figure 3-15) used in the compression tests is a tabbed flat coupon specimen with a short untabbed central length of 1/8-inch. The bonded tabs are released from the specimen in the central one-inch portion by Mylar. Support in the tab section is provided by clamping with heavy steel blocks which are bolted together. Comparison of the results from "Columbus" type specimens with those obtained on lateral leaf supported specimens indicates that much higher RT results were obtained by the latter method of testing.

3.2 COVER SKIN PROPERTIES

Mechanical Properties

Results of tests on tensile and compressive specimens representing the 12 ply cover skin orientation, $(0_3/\pm45/90)_s$, are shown in Table 3-15. The compression specimens were run as honeycomb stabilized sandwich coupons in edgewise compression. This represents the actual configuration and normally also provides indirect information on the adequacy of the core-to-face bond.

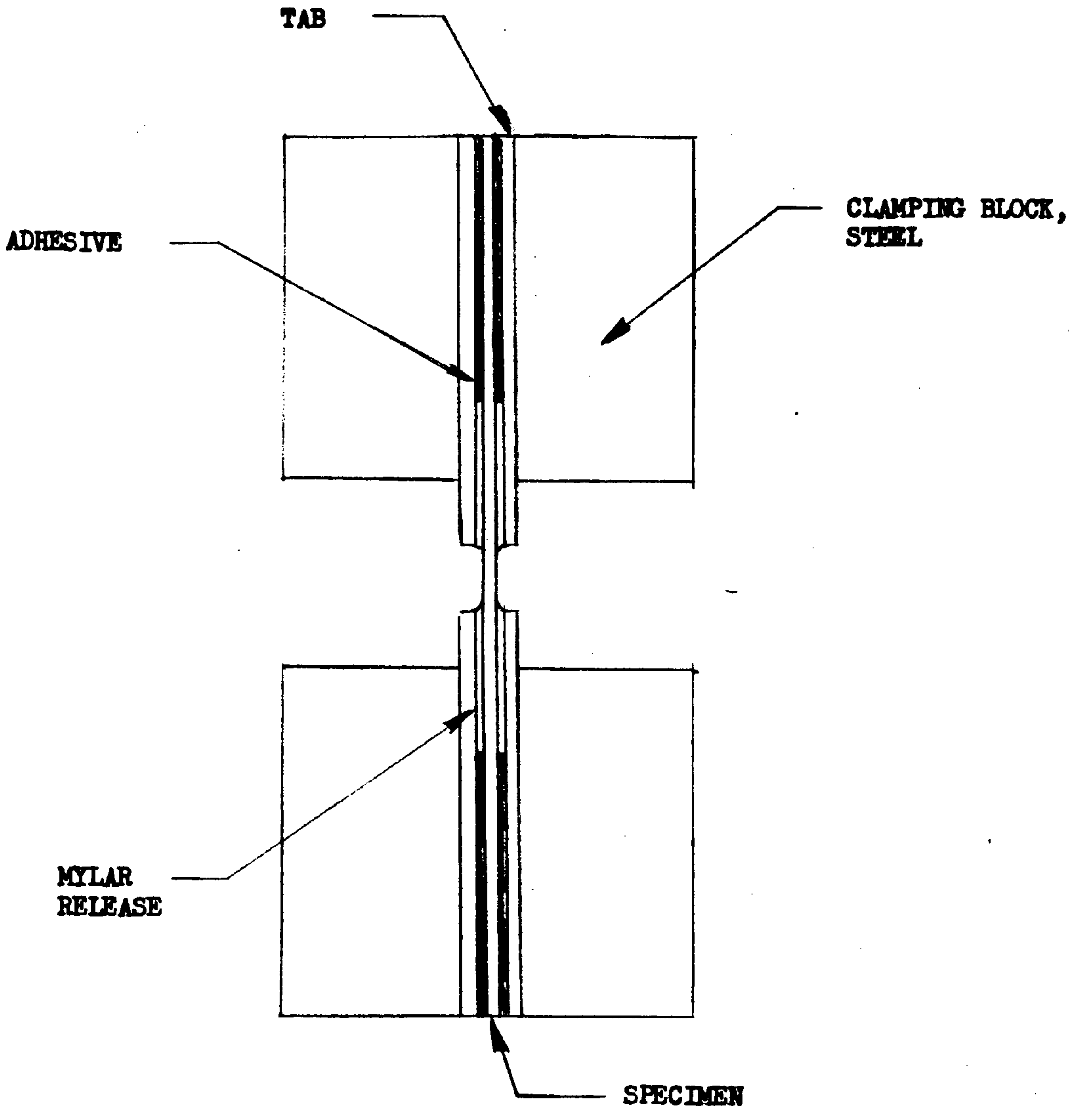


Figure 3-15. Columbus Compression Specimen Schematic

Table 3-14. Additional Compressive Data, Gemon L/Modmor II Composites

	RT	500 F After 1/2 Hr at 500 F	Panel and Remarks
Longitud compr str, ksi	61.22	68.75	PG 41-19-1
	85.82	62.95	"Columbus" Specimens
	81.35	62.37	
	76.13*	64.49*	
Transv compr str, ksi	16.41	12.74	PG 28-1-22
	13.32	11.67	
	17.57	13.79	
	15.79*	12.73*	
Mod, msi	1.19	0.89	
	1.05	0.87	
	1.26	0.78	
	1.7*	0.8*	

*Average

Table 3-15. Mechanical Properties of Cover Skin Arrays, $(O_3/\pm45/90)_s$

Test Temp	Panel PG 54-1B Tensile				Panel PG 54-1A-19 Compressive*			
	Strength, ksi		Modulus, msi		Strength, ksi		Modulus, msi	
	Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse	Longitudinal	Transverse
RT	74.8	48.4	12.4	5.2	58.1**	29.5	14.3	8.63
	74.8	40.9	15.0	5.1	54.9**		16.2	
	73.5	37.2	15.5	4.7	69.4			
	Av 74.4	Av 42.2	Av 14.3	Av 5.0				
500 F after 1/2 hr at 500 F	67.7	36.8	13.4		53.4	25.7		
	70.0	37.5	11.6		44.3	26.4		
	90.3	42.7	14.2			22.2		
	Av 76.0	Av 39.0	Av 13.1					

*Edgewise compression, H/C sandwich
**Single face failure, evidently due to imperfect alignment

However, the high sensitivity of longitudinal specimens to minor misalignments produced premature, single facing failures in most cases where the alignment had not been checked by strain gage outputs from both faces. Although the data generated is less useful than intended, it does show conformance with expected behavior. Therefore, no repeat testing was performed.

Thermal Expansion

The thermal expansion characteristics of Modmor II/Gemon L laminates were determined over the temperature range of -300 F to +500 F for a unidirectional array and one representing the cover skin array. Data on the laminates used for specimen preparation are as follows:

	PG 45-1-19A	PG 42-1-19A
Array	(O_{24})	$(O_3/\pm 45/90_2/x\ \pm 45/O_3)_s$
Density, lb/cu in.	0.0552	0.0545
Fiber volume, %	62.6	60.1
Resin content, % by wt	31.6	34.1

The specimens were tested per NR/SD Procedure No. 3340-002, "Linear Thermal Expansion—Dilatrometric Method." At low temperatures the test atmosphere was helium, while the high temperature dilatometer tube was purged with nitrogen gas. Heating rates varied from 2.8 to 3.6 degrees F/minute, and cooling rates, which are more difficult to control, ranged from 3.1 to 5.4 degrees F/minute.

Because of the very low expansivities of the materials, several steps were taken to ensure maximum accuracy. A fused quartz dilatometer was substituted for the Vycor unit used for previous high-temperature testing, the Vycor calibration having been found to be unstable. A Vycor unit was, however, used for the low-temperature tests. Both dilatometer tubes were recalibrated with a new OFHC copper standard.

The results, including estimated error, are:

Material	Test Direction	Temp Range, F	In./In. - F	Estimated Possible Error, %
PG 45-1-19A	Parallel	75-(-300)	-3.8×10^{-7}	±44
PG 45-1-19A	Parallel	75-500	8×10^{-8}	±221
PG 45-1-19A	Transverse	75-(-300)	1.5×10^{-5}	±2.4
PG 45-1-19A	Transverse	75-500	2.5×10^{-5}	±1.7
PG 42-1-19A	Parallel	75-(-300)	$<2 \times 10^{-7}$	-
PG 42-1-19A	Parallel	75-500	$<2 \times 10^{-7}$	-
PG 42-1-19A	Transverse	75-(-300)	8.6×10^{-7}	±24
PG 42-1-19A	Transverse	75-500	$<2 \times 10^{-7}$	-

It is apparent that the measurement error becomes excessive for the quartz or Vycor tube dilatometer system when the measure α is less than about 1×10^{-6} in./in. - F, for temperature ranges of the order involved here.

The data indicates that with a system of balanced plies of unidirectionally fiber-reinforced material, expansion coefficients are very near to zero.

3.3 FABRICATION PROCESS STUDIES

Effect of Fiber Choice on Ply Thickness

It had been observed in the resin and fiber selection studies that the laminate ply thickness of Gemon L/Modmor II material tended to be higher than that of the initially predominently used Gemon L/HTS material. It was decided to accept the higher ply thickness of the Modmor II system rather than attempt to correct it by a downward adjustment of the resin content of the prepreg. Such an adjustment of the prepreg resin content could result in processing difficulties and would have required additional fabrication process studies.

Heating Rate

The initially produced Gemon L/Modmor II panels of the fiber selection studies (Set 1 of Table 3-10) had also exhibited a substantial spread in ply thickness.

Photomicrographs (80X magnification) of the unidirectional panels produced in this task were taken in an effort to learn whether a correlation could be established between heat-up rates during cure and the void content and microstructure. As evident from analysis of Table 3-10 and the microstructures of the various panels as shown in Figure 3-16, no straight-forward correlation could be established.

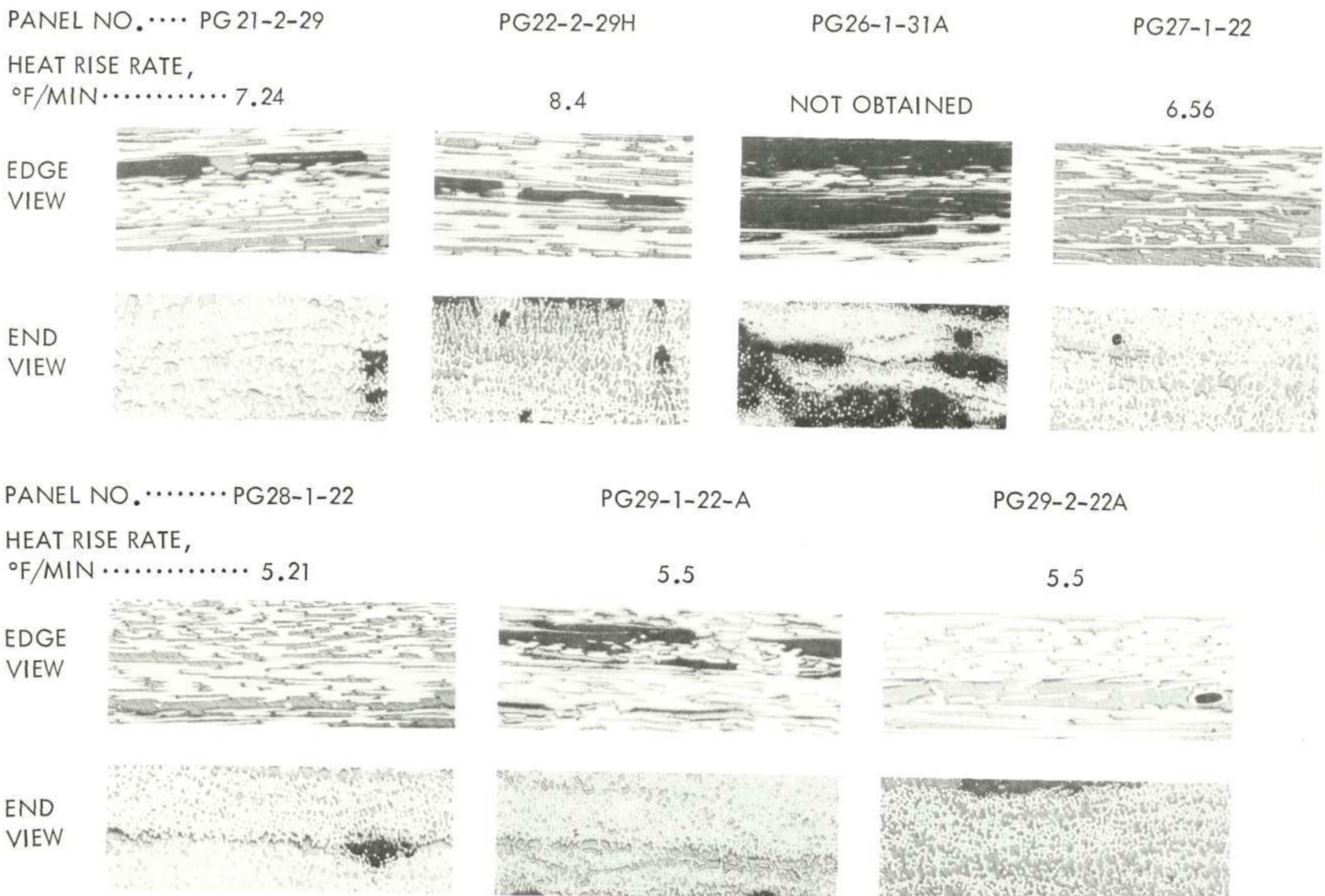


Figure 3-16. Typical Microstructure of Unidirectional Gemon L/Modmor II Composites

Another concern regarding heating rates was that, due to the mass of tooling required, the tentatively identified process requirement of 5.0 to 6.5 F/minute could not be met in production. The mass of the tooling for fabrication of the box beam elements presents a possible problem in attaining this desired heating rate. A laminate fabricated at a heating rate of 2 F/minute under production conditions resulted in an unacceptable ply thickness of 6.6 mils. Laboratory produced panels at 2 F/minute heating rates produced similar thicknesses; see Set 2 of Table 3-10.

A heating rate of 3-5 F/minute was eventually established as acceptable and realizable in production equipment by trial runs on Gemon L/Modmor II material purchased to the requirements of the Material Specification, Appendix B. The following equipment modifications were made on the 5-foot diameter x 10-foot long Baron autoclave to permit attainment of higher heating rates.

1. Change of the CO_2 pressurization system line from 3/4-inch to 2-inch diameter to increase pressure rise.

2. Reduction in the support plate thickness to reduce heat sink.

3. Insulation of the tooling and component from the handling equipment.

Bleeder and Post Cure Schedule

Flexural and short beam shear tests were performed on specimens from Gemon L/Modmor II panels prepared to assess changes in composite properties due to the following variations in processing procedures:

1. Utilization of top and bottom bleeding in lieu of only top bleeding.

2. Shortening of the post-cure cycle from a total of 108 hours to 28 hours (10 hours from 300 F to 500 F and 18 hours at 500 F).

Top bleeding only gave poor results for integrally molded/bonded joints simulating the titanium end fitting to composite skin joints. Top and bottom bleeding upgraded joint performance considerably (see Section 4.1) due to venting of volatiles which otherwise would be trapped between the lay-up and the lay-up plate. Shortening of the post-cure cycle is highly desirable from a production standpoint.

The results, shown in Table 3-16, indicate slight reductions in properties because of the measures taken when compared with previous data. However, the advantages of these procedures were judged to outweigh these effects. Top-and-bottom bleeding and the 28-hour shortened post-cure were incorporated as process parameters for pertinent article details.

Table 3-16. Effect of Bleeder and Post-Cure Variables on Unidirectional Properties of Gemon L/Modmor II Composites

Panel No. and Process Variable	Flexural Strength, ksi		Flexural Modulus, 10^6 psi		Short Beam Shear Strength, ksi	
	RT	500 F After 1/2 Hr at 500 F	RT	500 F After 1/2 Hr at 500 F	RT	500 F After 1/2 Hr at 500 F
PG 33-1-19 Top and bottom bleed; 108-hr post-cure	185.5 190.0 173.1 182.6 Av	129.1 140.1 136.9 135.3 Av	13.8	10.7 12.7 12.0 11.8	9.99 9.65 10.59 10.07 Av	7.14 6.52 7.53 7.10 Av
PG 36-1-22 28-hr post-cure	190.9 180.4 205.7 192.3 Av	129.1 150.5 150.6 143.6 Av	15.9 15.4 17.0 16.0 Av	11.5 15.1 13.3 Av	11.39 12.50 10.37 11.42 Av	6.90 5.40 7.20 6.50 Av
PG 36-2-22 28-hr post-cure	209.8 213.8 211.8 Av	111.4 145.4 128.4 Av	15.8 15.2 15.5 Av	12.9 14.1 13.5 Av	10.98 12.69 11.84 Av	7.00 7.50 6.85 7.12 Av

Reference is made to Table 3-10 for physical properties of the composite panels used in the described test series.

Perforated Aluminum Caul Plate Studies

The use of a perforated caul plate to insure adequate venting is considered necessary for fabrication of panels of large area. Thus a study was instituted to determine the feasibility of this approach. A Gemon L/HTS panel was fabricated in accordance with Process 15 and utilizing an aluminum caul plate perforated with 0.06-inch diameter holes spaced on 1.0-inch centers. The top-and-bottom "bleeder-breather" systems were interconnected. No mark-off from the holes in the caul plate was observed on the finished panel. The RT flexural test results shown on Table 3-17 are in agreement with data obtained from panels using non-perforated caul plates. It should be noted that specimens PG 20-1-22-1A, -2A, and -3A were tested as-molded while specimens PG 20-1-22-4B, -5B, and -6B were ground to a flatness tolerance of ±0.0005 inches to determine the effects on property values of cutting 0° fibers. The test results showed no effect from the cutting operation.

Table 3-17. RT Flexural Test Results HTS/Gemon L Panels Molded Per Process No. 15 With Perforated Caul Plate

Specimen No.	Thickness, Inches	Flexural Strength ksi	Flexural Modulus 10^6 psi
PG 20-1-22-1A	0.075	206.0	18.1
-2A	0.070	200.2	17.6
-3A	0.074	200.1	16.4
		202.1 Av	17.4 Av
PG 20-1-22-4B	0.065	200.0	16.5
-5B	0.064	212.1	16.7
-6B	0.064	211.1	17.9
		207.7 Av	17.0 Av

Bagging Studies

Dummy runs at temperature were planned for rib-to-spar subassembly and the final assembly bonding. Furthermore, the set-up for both operations was visualized to be essentially identical. Estimates indicated that a reusable silicone rubber bag would be more economical than conventional PVA or nylon film bagging for each operation. Also, far greater confidence could be placed in the reliability of its performance. The basic design approach for the bag is shown in Figure 3-17.

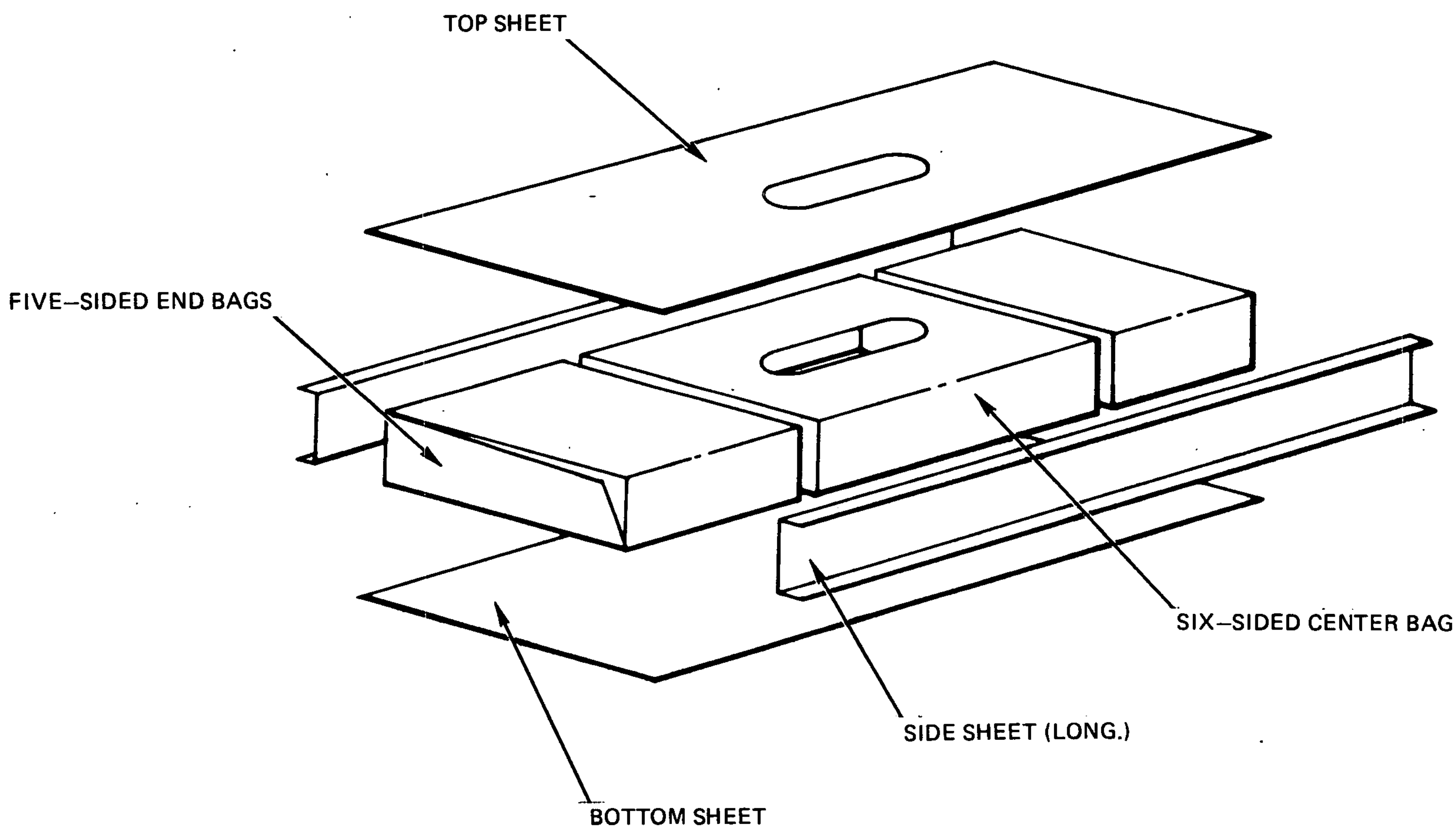


Figure 3-17. Vacuum Bag for Graphite-Polyimide Box Beam Assembly Bonding

Two types of high temperature silicone rubber sheets, Connecticut Hard Rubber Co. CHR 9255 (38 inches wide) and CHR 207 (48 inches wide), were considered as candidates. Empirical tests were conducted to determine their use limitations as follows:

1. Exposure to 365 F under 75 psi pressure for six hours.
2. Compatibility with sealant putties at 365 F for six hours.
3. Determination of bond strength with RTV 102 adhesive after multiple cure exposures (approximately five times).

The criterion for determining the gross effect of the exposure conditions was manual notch-tear testing of the silicone rubber. Silicone rubber will tear or break easily if any reversion or degradation has taken place.

Both materials were satisfactory under these test conditions. Joints contemplated for the sections of the rubber bag then were evaluated. The removable joint (between the top sheet and the remainder) would employ zinc chromate sealing putty. Compatibility of the sealant putty with the rubber at 365 F for six hours was verified. The permanent joints were to be made with RTV 102 adhesive. Compatibility tests were run for five cure cycles. The RTV 102 caused a breakdown of the CHR 9255 rubber and exhibited low strength (poor peel resistance) with CHR 207. Since both end bags and the center bag were nearly completed by the time this test was finished, they were scrapped.

Two other adhesive systems were evaluated (DC 92-018 and DC 93-046). One of these, DC 92-018, proved very compatible with the silicone rubber and retained high peel strength. CHR 207 rubber joined with DC 92-018 showed no detectable degradation when tested at five cycles to 400 F (including 52 hours exposure at that temperature) followed by one cycle to 500 F (with a 14-hour hold at that temperature). Since this is a far more severe exposure than the bag would see in service, this system was qualified for use. The bag elements were fabricated using DC 92-018 adhesive and CHR 207 rubber. The bag was tested for leakage on the dummy tool and showed no evidence of leaking even with the vacuum pump disconnected over a weekend. The static gage indicated a 26 in. Hg vacuum.

3.4 MATERIAL PROCUREMENT SPECIFICATION

The Gemon L/Modmor II Procurement Specification to which production material was purchased is presented in Appendix B, together with a description of the testing procedures used. This version reflects adjustments, as dictated by experience, from "best guess" stipulations during early portions of the program, and agreement by the vendor, General Electric, with respect to realism of posed requirements (values and tolerances) as well as test methods.

Particularly noteworthy is the fact that the specification controls the areal weight of the graphite fiber reinforcement. This feature, in NR's opinion, is extremely helpful in providing downstream weight and performance control.

Load carrying capacity and stiffness characteristics at any locale are primarily functions of amount of fibers and, of course, their orientation. These parameters at any section are controlled by specifying them in terms of number and orientation of plies. Deviations in amount of fiber per unit area of ply from the values and/or tolerances assumed in design considerations will have large effects on actual versus predicted performance. Since only a portion of the plies run in the same direction in the multi-directional arrays most often used in aerospace composite structures, the averaging effects between local deficiencies of parallel plies are far lower than those in conventional parallel laminated glass fabric reinforced plastic structures.

4.0 ADHESIVE BONDING

FM-34, Bloomingdale Department of American Cyanamide Corporation, was selected for all adhesive bonding operations, i.e., co-cure with Gemon L/Modmor II prepreg if required, secondarily bonded continuous surface joints, and honeycomb core-to-face joints, for the following reasons:

1. Usage in the related NR/COL B/PI Wing Box Beam program (no or little development effort requirements anticipated).

2. Usage at NR/LAD (Reference 6), and at NR/SD in bonding of insulation to the Apollo spacecraft launch escape tower.

3. Choice in the then ongoing Boeing SST Program with attendant substantive literature information.

4.1 CO-CURED JOINTS

Orientation tests, titanium-to-composite, were conducted on 1/2 inch overlap, double lap joint specimens, representative of the titanium end fitting-to-composite laminate joints. The titanium adherends were primed with Bloomingdale BR-34 primer. Poor results were obtained with integrally cured bonds using Process 15 parameters.

The observed warpage and post-test bondline conditions revealed that the uneven, top only, bleeding technique used was not satisfactory. They also indicated that co-cure with FM-34 would be desirable in view of the low transverse strength of the Gemon L composite. This deficiency was evidenced in these studies by a tendency toward longitudinal hairline splitting during post cure.

Integral bonding studies of Gemon L/Modmor II prepreg to titanium were conducted with emphasis on the use of very thin (nominal 0.03 psf) co-cured FM-34 adhesive. Douple lap, 3/4 inch, tensile shear specimens averaged 2538 psi at RT and 1457 psi at 500 F after 1/2 hour at 500 F, see Table 4-1. These specimens were fabricated using Process 15, with a shortened post cure, and utilizing a top and bottom bleeder system. Measured glue line thicknesses were in the 2 to 2.5 mil range. This is far more desirable than the approximately 4 to 4.5 mil glue line produced by 0.06 psf adhesive if the available cross-section of the step lap titanium end fittings is not to be unduly reduced.

Table 4-1. Tensile Shear Strength, Integrally Cured Joints Gemon L/Modmor II Composite to Ti-6AL 4V FM-34 Adhesive, 0.3 psf Double Lap Shear Specimens, 3/4-Inch O. L.

	Lap Shear Stress at Failure, psi	Mode of Failure			
		Primer (%)	Bond (%)	Composite (%)	Titanium (%)
RT	2767		50	50	
	2367				100
	2597	50	50		
	2590				100
	2367				100
	2538 Average				
500 F after 1/2 hour at 500 F	1383	25	25	25	
	1507	50	50		
	1347	30	50	20	
	1517	30	50	20	
	1530	40	40	20	
	1457 Average				

The adequacy of the co-curing approach was further verified by structural element tests, Section 6.1.

4.2 SECONDARILY BONDED CONTINUOUS SURFACE JOINTS

Cure Cycle Studies

The G/PI Center Wing Box Beam design calls for a number of adherend combinations in secondarily bonded continuous surface joints, the most critical being the glass fabric/PI laminate spar lap joints to the mating G/PI and titanium end fitting surfaces of the top and bottom cover panels. Secondary bonds between Gemon L/Modmor II laminate and cured 35-512 glass fabric/4707 laminate using FM-34 adhesive, 0.135 psf, cured under 3 psi dead weight load at 350 F for 2 hours and with the Gemon L shortened post-cure (28 hours total with 10 hours at 500 F) resulted in low bond strength values both at RT and 500 F, see Table 4-2. The bonding condition simulated the one originally planned for final assembly of the upper and lower panels to the spars and ribs, utilizing the B/PI box beam tooling and load conditions identical to the ones then planned for it.

Table 4-2. Tensile Shear Strength, Secondary Bonds*
Pyralin 35-512 Laminate/Gemon L - Modmor II
Composite FM 34 Adhesive, 0.135 psf

Double Lap Shear Specimens, 1.0-inch O.L.

Test Temperature	Tensile Shear Strength, psi* — Dead Weight Load Bonding Pressure: 3 psi**	Tensile Shear Strength, psi* — Dead Weight Load Bonding Pressure: 10 psi***
RT	300 380 263 314 Average	534 684 651 623 Average
500 F after 1/2 hour at 500 F	513 443 580 479 Average	

*3.9 F/minutes, heat rise to 355 F, hold for 120 minutes at 355 F. Shortened Gemon L/Modmor II post-cure (28 hours total with 10 hours at 500 F).
**All failures adhesively from 35-512 laminate.
***All failures cohesive in bond line.

A second test series was performed at a dead load condition of 10 psi simulating the pressure obtained from an increase in dead weight tooling load capacity to 4500 to 5000 pounds over the planform area of the cover panels. The results, also shown in Table 4-2, were still quite low and a matter of concern even though they are about double those obtained with the 3 psi cured joints. This concern was related to NASA/MSFC and tooling methods for obtaining higher pressures (40 psi minimum) were considered. However, no immediate action was initiated since design requirements were met by the values of the 10 psi pressure bonded specimens.

The B/PI box beam failed to meet design expectations in structural test June, 1971, the bond line between one of the spars and the upper cover failing prematurely. This led to a decision to design and fabricate a special tool, capable of applying at least 55 psi bonding pressure. Ability to draw off volatiles generated during cure was considered in choosing the approach and tooling concept, but no special provisions were made for the following reasons:

1. Fabrication of components along originally planned design lines had commenced and progressed to a state not permitting design changes.

2. The design concept had not envisioned bagging procedures to be used in adhesive bonding assembly of the component members. The reliability of such procedures for the existing design was considered questionable due to the accessibility difficulties and bag complexity connected with the I-shape cross-section of the spar and rib members and the sectioned-off design of the assembly.[1]

3. Conversations with Bloomingdale, Boeing, and Heath-Techna personnel elicited in each case identical opinions that side venting from the maximum 2-1/4 inch wide glue lines should be adequate and that no vacuum provisions should be necessary. Increase in positive pressure to a minimum of 35 psig was considered necessary, however.

Disappointingly low results were obtained on in-process Ti-to-Ti control specimens in initial trial runs of the new tool in the spar-to-rib assembly mode. At first, it was assumed that improper application of pressure was responsible for the poor results; several tooling modifications were made to correct the cause of the suspected problem. Since an apparent improvement was noted, it was decided to evaluate the adhesive. In a meeting with Bloomingdale, the vendor indicated that moisture pick-up by the adhesive might well be the reason for poor test results and that premature opening of the sealed package upon removal from the 0 F storage refrigerator could easily provide a moisture source.

Samples of the material in storage at NR/SD (Batch 139, Roll B314) were forwarded to Bloomingdale for evaluation. The results of testing by the vendor are presented in Table 4-3.

It becomes apparent on examination of this data that the material had indeed been affected by moisture and would not produce satisfactory results (1200 psi at 500 F) even with proper application of pressure. It also indicates

[1]The validity of this concern was borne out by bag seam failures in later assembly trial runs, see Section 7.1.

Table 4-3. Results of Bloomingdale Investigations of FM-34 Adhesive

Batch and Mfg Date	139; January 20, 1971		143; August 26, 1971	
Roll	B314		B321	
Reconditioning	None	48 Hr RT Dririte	Not Required	
Curing pressure	50 psi	50 psi	50 psi	50 psi + vac
Heat rise rate	1.5 F/min	1.5 F/min	1.5 F/min	60 min to 350 F
Hold time at 350 F	2 Hr	2 Hr	2 Hr	3 Hr
Post cure	3 Hr at 400 F+ 4 Hr at 500 F	3 Hr at 400 F+ 4 Hr at 500 F	3 Hr at 400 F+ 4 Hr at 500 F	Oven at 350 F raise to 550 F, hold 3 Hr
Lap shear strength, R.T.	1615, 1615	1920, 1976	2020, 1820	3800
Lap shear strength, 500 F	1010, 755	1665, 1110, 1550	1565, 1515	2000

that reconditioning with a desiccant can essentially restore the material to a satisfactory condition. Batch 143, Roll B321 was newer material on hand at Bloomingdale which was also tested for comparison with the material from NR/SD. As can be seen from the data in Table 4-3 the reconditioned Roll B314 and Roll B321 materials produce very similar results when processed under the same conditions. Roll B321 material was also tested using a vacuum and a higher heat rise rate in processing. Use of this cure cycle produced considerably higher results than achieved in the previous specimens. The vendor claimed that the increased properties were due primarily to the increase in heat rise rate, not the vacuum application. Additional bonding studies were carried out using FM-34 adhesive cured under several processing conditions; the results are reported in Tables 4-4 through 4-6. While the studies were not extensive enough to develop statistical conclusions, several general observations are possible.

1. Moisture pick-up is possible if the material is not stored with a suitable desiccant or if insufficient warm-up time is allowed before opening the bag seal on the adhesive tape which was stored at 0 F refrigeration. Pick-up of moisture is detrimental to the FM-34 adhesive system.

2. Moisture can be removed from the adhesive by storage with a suitable desiccant, bringing it back to a usable condition.

3. Both moisture and some of the other volatiles can be extracted by staging at 225 F for 30 to 40 minutes, but staging for 3 hours at 225 F appears detrimental to the high temperature properties of the adhesive system. Staging results in a "boardy" product which is considerably harder to work with than unstaged material.

4. Higher heat rise rates appear to produce better properties in adhesive cured with positive pressure only, but is not as important when curing is carried out using a vacuum in conjunction with positive pressure.

5. Entrapment of volatiles is a primary cause of low bond strength with FM-34 adhesive, and larger bond areas increase the difficulty as shown by the difference between single lap specimens employing no shims and those with additionally bonded shims. Vacuum application during cure aids considerably in volatile release and results in substantially increased bond strength.

From this study series it was concluded that the use of positive pressure only on well desiccated or staged material would probably result in marginal but adequate bond strength in the joints. The use of vacuum in addition to positive pressure would provide a much higher degree of confidence in the adequacy of the secondarily bonded joints of the box beam assembly.

Table 4-4. Initial FM-34 Adhesive Studies

Adherends	Adhesive	Condition	Specimen Configuration		Cure Pressure		Heat Rise Rate Deg F/Min	Test Results	
			A	B	Positive Psi	Vacuum		R. T.	500 F
Titanium to Titanium	Batch 143 Roll 321	H_2O Exposure	X		50	No	2	906	663
		Desiccated	X		50	Yes	2	2890	1508
		Desiccated	X		50	No	2	777	795
		Desiccated	X		50	No	4.5	1426	989
		Desiccated	X		50	Yes	4.5	3310	1830
		Desiccated		X	70	No	2		1109
Titanium to Titanium	Batch 139 Roll 314	H_2O Exposure		X	70	No	2	Fell apart	
		Staged, 30 min., 225 F		X	70	No	2	1560	
		H_2O Exposure	X		70	No	2		1513
		Staged, 30 min., 225 F	X		70	No	2		2197
		Staged, 30 min., 225 F	X		70	No	2		1785
		Staged, 30 min., 225 F	X		70	No	4.5	1680	1164
		Staged, 30 min., 225 F		X	70	No	4.5	1543	1195
		Staged, 3 hr, 225 F		X	70	No	4.5	1480	567
Glass/PI to Graphite/PI	Batch 103 Roll 321	Desiccated	X		50		2	1064	326
		Desiccated	X		50		4.5	1572	729
		Desiccated	X		50	Yes	4.5	2713	1589

A - Single Lap
B - Single Lap with Full Shims

Table 4-5. Overview, Effects of Cure Cycle Parameters

Secondary Bonds, FM-34, Ti - TO - Ti

Applied Pressure		Vacuum in. Hg	Heat Rise Deg F/Minute	Intentional Questionable Handling	Av Shear Strength, psi	
Psig	Time				RT	500 F
50 - 70	Start	None	2	Yes	0 - 906	663
50 - 70	Start	None	2	No	777 - 1560	795 - 2197
50 - 70	Start	None	4.5	No	1426 - 1680	567 1195
50	300 ± 30 F	25 min	2	No	2890	1508
50	300 ± 30 F	25 min	4.5	No	3310	1830
55	Start	5	3.2	No	850	604
55	Start	15	3.2	No	837	1337
55	Start	25	3.2	No	2820 - 949	1490 - 660
55	Start	25 min	4	No	3027	1532
55	270 - 330	25 min	2 - 4	No	2459 - 3315	1585 - 1768
None	-	25 min	4	No	3143	1678

Table 4-6. Secondary Bonds, FM-34 Wing Box Adherend Combinations

Applied Pressure		Vacuum In. Hg	Heat Rise Deg F/Minute	Adherend* Combination	Av Shear Strength, Psi	
Psig	Time				RT	500 F
3	Start	None	3.9	GL/GR	314	479
10	Start	None	3.9	GL/GR	623	-
50	Start	None	2	GL/GR	1064	326
50	Start	None	4.5	GL/GR	1512	729
50	300 ± 30 F	25 Min	4.5	GL/GR	2713	1589
70	Start	None	2.2	TI/GL	1430	1210
70	Start	None	2.2	GL/GR	780	670

*GL = Glass fabric/PI; GR = graphite/PI; TI = Titanium

In a December 1971 meeting in Huntsville, Alabama, NASA/MSFC was advised in detail of the technical findings as well as the budgetary status of the program, including expenditure of company funds in an attempt to achieve satisfactory completion of the G/PI Center Wing Box Beam Program. After resolution of funding and schedule adjustments, pursuit of the program was resumed in April 1972, with redirection to employ vacuum bagging measures as required to produce reliably high quality joints.

A number of additional studies were performed, aimed at disclosing the effect of the following variables:

Vacuum level
Applied pressure level
Timing of positive pressure application
Effect of questionable handling
Heat rise rate during cure
Joint width
Glue line thickness
Assembly aid fasteners
Adherend combination
Repriming procedures

Table 4-5 presents an overview of the results of the cure cycle parameter directed studies, performed on Ti-to-Ti 1/2-in. O. L. specimens. It is apparent that maintenance of a rather high vacuum (25 in. Hg is satisfactory, but 15 in. Hg was not) during FM-34 adhesive cure is the single most important feature in reliable and reproducible attainment of high bond strength levels. Applied pressure level and timing, heat rise rate, etc., can be varied over rather wide ranges without significant effects.

Results of tests on specimens representing the major wing box adherend combinations are summarized in Table 4-6. Again, only the specimens bonded with the aid of 25 in. Hg vacuum during cure developed bond strengths meeting the new acceptability criteria.

Geometry Variables

Effects of geometry variables also produced only minor effects. Specimens simulating the 2-1/4 inch spar-to-cover joint width were comparable in strength to 1-inch wide control. The tensile shear strength of 2-1/4 inch wide ti-to-ti coupons, 1/2 in. O. L. is shown below:

RT	500 F After 1/2 Hr at 500 F
3478 psi	1764 psi
3001 psi	1823 psi
3240 psi Av.	1794 psi Av.

With respect to glue line thickness, study results (Table 4-7) indicate that the strength of the controls was maintained for thicknesses up to 0.030 inches at least, provided the number of adhesive layers is reasonably matched to the expected gap dimension. The latter, in the case of actual assembly operations, is established by pre-fit impression check runs.

It was anticipated that use of a vacuum bag would make it difficult to index and insure positive part location on the existing bonding fixture, since the bag hides the coordinating features of the tool. Assembly aid fasteners therefore were considered necessary. They should be easily reusable and removable since dummy runs were to precede actual assembly bonding operations and they should be able to accommodate the dimensional changes caused by differential thermal expansion of mating parts during cure. If mechanically locked together at room temperature, improper geometry and flow conditions would prevail at the glue line at the setting temperature of the adhesive, impeding correct movement at that time.

A laboratory test was set up in which three types of specimens were evaluated, using 1- by 6-inch strips of 0.062-inch thick aluminum bonded with FM-34 to 0.032-inch thick titanium as follows:

1. No mechanical fasteners.
2. Dowel fasteners, 1/8-inch diameter, on five-inch centers.
3. Bolts and nuts joined on five-inch centers with slotted holes and teflon washers.

Table 4-7. Effect of Glueline Thickness, FM-34 Adhesive, 0.135 PSF

No. of Plies Adhesive	Glueline Thickness, Mils		Av Lap Shear Strength, Psi
	Intended	Actual	
1	Control	10.0 - 12.0	3436
	7	9.0 - 13.0	3663
	10	11.5 - 16.0	3738
	15	15.0 - 21.0	2971
2	10	14.0 - 15.0	3818
	15	13.0 - 18.0	3418
	30	20.0 - 32.0	2217
3	20	19.0 - 25.0	3349
	30	23.0 - 30.0	2945
	40	30.0 - 36.0	2156
2 plus 0.010" Solid Shim	24	27.0 - 29.0	2955*
	30	30.0 - 33.0	3019*

*Failed in glass fabric laminate shim

The slip washer/bolted assemblies developed essentially identical residual thermally induced mechanical stresses as the unrestrained ones, as evidenced by the similar curvature of the specimens after bonding. The curvature of the ones pinned with dowel pins was much greater. The assembly aid concept of bolting with the aid of slotted holes and Teflon slip washers was incorporated into the wing box design. Original plans to use commercial slip fasteners such as Tinnerman Tubular Clips or Carr Palnut fasteners were discarded because of probable inavailability in time to meet schedule.

Reconditioning of Titanium Surfaces

The titanium end fittings on the cover plates had been primed approximately 12 months prior to actual use. The quality of the "aged" primer was unknown. Therefore, a procedure was worked out to strip and reprime local areas. Two methods of surface reconditioning were investigated. One method uses only abrasive cleaning and the other both an abrasive plus Pasa-Jell 107 surface treatment. Titanium test coupons were primed with BR-34 primer and cured per MB0120-062. The primer was stripped and then the coupons were reprimed. Lap shear specimens were bonded with FM-34 adhesive per MB0120-062 and tested at both room temperature (RT) and at 500 F (after one-half hour exposure).

Average test values were as follows:

Reconditioning Method	RT	500 F
Abrasive	2672	1547
Abrasive plus Pasa-Jell 107	2913	1794

These results indicate that either method is satisfactory based upon target values of 2500 psi at RT and 1200 psi at 500 F.

The procedure for the assembly, Appendix C, Table C-2, calls for abrasive cleaning, careful masking, and local manual surface preparation with Pasa-Jell 107.

Production Equipment Modification

Heat rise rates were studied in the large Lacy autoclave used for Apollo bonding operations. This autoclave had been identified as the scheduled heat source, but not as the pressure medium in the assembly bonding operations. The primary pressurizing approach, for reliability reasons, is via the bonding tool. Heat rise rates were determined to be satisfactory, but special ducting had to be installed to produce more uniform heating over the complete assembly. This was especially true for the central interior portion.

4.3 SANDWICH BONDING

Due to the high volatile release encountered in curing FM-34 adhesive, 2-foot by 2-foot aluminum sandwich panels were prepared using perforated and non-perforated 1/8-inch cell size aluminum core. Bonding was accomplished with 0.135 psf FM-34 supported film adhesive on core roller coated with BR-34 primer in accordance with dried weight parameters developed by Boeing for SST applications. Fabrication of these panels served a dual purpose:

1. Familiarization of personnel with roller coating procedures.

2. Establishment of the effects of high gassing tendencies on the bond strength of the system.

During trimming operations large amounts of gas were released from the panel with non-perforated core. The results of RT flatwise tensile testing, Table 4-8 indicates that trapping of these gases during cure does reduce core-to-face bond quality.

Table 4-8. RT Flatwise Tensile Strength Aluminum Honeycomb Core, 1/8-in. Cell-to-Aluminum Facing

RT Flatwise Tensile Strength, psi	
Perforated Core	Non-Perforated Core
835 743 835	580 594 621
804 Av.	598 Av.
All core tension failures	All skin-to-core failures

*Cured per the following schedule:

1. Apply full vacuum (26 in. Hg.).
2. Raise panel temperature to 325 F at a heat rise rate of 3.9 F/min in autoclave.
3. Apply 55 psig within 2.5 minutes.
4. Continue heat rise at 3.9 F/min to 350 F. Cure for two hours.
5. Post cure unrestrained four hours each at 400 F and 500 F.

Although the relatively high porosity of HRH-327 core* cell walls should greatly reduce the severity of this problem, information was sought on the possible reduction in properties caused by slotting the cell walls. Such grooving would provide an interconnection between the cell walls, thus creating a path for ready escape of gases produced during cure of the adhesive. Slotting of the cell walls was 1/8" deep at a 30° angle to the ribbon direction of the core. Bonding of the core to the BR-34 primed aluminum faces was as follows:

1. Roller coat core on both sides with BR-34 primer (81% solids). Air dry for 30 minutes and force dry for 30 minutes. Final dried weight of the applied primer shall be 27 to 32 gm/sq ft (both surfaces).

*Called for in the wing box design

2. Apply FM-34 adhesive, 0.135 psf, to skins.

3. Cure according to the following schedule:

   a. Apply 25 inch hq vacuum

   b. Raise temperature to 325 F at a rate of 4 F/minute

   c. Apply 55 psi autoclave pressure

   d. Continue raising the temperature to 350 ± 10 F at 4 F/minute

   e. Cure for two hours. Cool under pressure to 150 F and remove from autoclave.

Except for the four-hour hold at 400 F in lieu of three hours, this cycle is identical with the one for the cover and spar sandwich assemblies. Short beam sandwich shear specimens (core ribbon direction parallel to the major axis) were tested in flexure. The results, shown in Table 4-9, confirm that mechanical properties of the core are not adversely affected by slotting and that slotting is a feasible gas venting approach for this type of core.

Skin-to-core specimens with (± 45) orientation also were processed as described above and tested in flatwise tension. The results, as shown in Table 4-10, indicated that the core-to-face bond is stronger than the interlaminar bond between the facing plies for both the slotted and unslotted configurations.

4.4 INTERPLY BOND STRENGTH

The interply bond strength of Gemon L/Modmor II composite and 35-503 (Pyralin 4707/181 glass fabric) laminate were determined per Federal Standard 406, Method 1042A (opposed notch shear strength), with the following results:

Material	Array	Average Shear Strength, psi RT	Average Shear Strength, psi 500 F
Gemon L/Modmor II	$(O_3/\pm45/90)_s$	1820	1807
35-503	Parallel	2010[1)]	1313[1)]

[1)] All tensile failures

Table 4-9. Effect of Slotting on RT Core Shear Properties,*
HRH-327-3/16-6.0 Core

Slotted	Core Shear Stress at Failure, psi	Computed Core Shear Modulus, ** ksi
None	424 432 467 Av 441	28.0 34.9 40.1 Av 34.4
One Side	494 428 362 Av 428	38.2 32.4 31.6 Av 34.1
Both Sides	433 434 351 Av 406	36.2 33.3 29.7 Av 33.1

*Short beam flexure, core ribbon direction parallel to major specimen axis.
**No correction made for facings.

Table 4-10. Flatwise Tensile Test Results, HRH-327-3/16-6.0
Core to (± 45) Gemon L/Modmor II Facings,
FM-34, 0.135 psf Adhesive

Core Slotted	Temp	Flatwise Tension Stress at Failure, psi*
No	RT	567, 580
	500 F	109, ** 320
Single Side	RT	568, 562
	500 F	360, 464

*With exception shown, all failures between outer and remaining plies of facing laminate.
**Skin-to-test block failure.

5.0 DESIGN

The graphite/polyimide box beam design was based on designs of boron/epoxy and boron/polyimide box beams (References 3 and 4). Since the purpose of the study was to establish the state-of-the-art in the manufacture of polyimide structures rather than to develop design and material data, design modifications were minimized. Some design modifications were performed, however, to account for new laminating and secondary bonding materials. Modifications also were necessary at positions where design philosophy had changed since the design of the boron/polyimide beam, namely in the titanium end fitting configurations and in the cover skin laminate ply orientation. In summary, the principal design changes were as follows:

1. Ply orientation
2. Titanium end fitting
3. Spar-to-cover joints

The design drawings for the graphite/polyimide box beam are presented in Appendix D. Dimensions were adjusted so that the outer envelope could be held to that of the boron/polyimide box beam (Reference 4) for use of existing test fixtures.

5.1 PLY ORIENTATION

Previous composite box beam designs employed a laminate configured by a computerized optimization procedure. The developed laminate consisted of ten plies of boron polyimide (+5./-12.5/+25./-40./+45./-45./+40./-25./+12.5/-5.) balanced across the honeycomb sandwich of the cover. Each ply was approximately 5.25 mils thick, resulting in a ten-ply skin gage of 0.0525 inches. Analysis of this laminate revealed that it had a relatively high Poisson's ratio (0.83) and became matrix dependent for biaxial load ratios other than the ratio for which it was optimized.

The NR design philosophy for structures similar to the box beam of this program has changed since the fabrication and test of the previous beams. This new philosophy limits laminates to ply combinations in directions defined as spanwise, chordwise and ± 45 degrees to the span. This 0/90/±45 pattern permits the distribution of a major portion of the spanwise, chordwise, and in-plane shear forces to specific layers. Therefore, the spanwise plies take the spanwise loads by virtue of their relatively high moduli. Similarly the ±45 degree and chordwise plies react the shear and chordwise loads, respectively. The presence of the chordwise plies also reduce the Poisson's ratio of the laminate to a value approximately equal to that of metallic materials. The separation loads to specific plies based on ply rigidity reduces the role of the matrix in the laminate. This factor is of special importance when the direction of principal forces varies in service, especially under elevated temperature conditions.

The graphite polyimide laminate cover skin pattern selected for this beam was based on matching of boron polyimide and graphite polyimide cover skin longitudinal-to-shear strength ratio and maintaining a minimum number of transverse plies. The selected $(O_3/\pm45/90)_S$ 12-ply laminate is compared to the previous boron polyimide laminate in Table 5-1. Since the thickness of these two laminates differ, both the rigidities and mechanical properties are listed. As shown in the table, the spanwise rigidity and tensile strength of the graphite polyimide laminate are approximately 90 percent and 85 percent, respectively, of the boron polyimide laminate. In addition, the Poisson's ratio has been lowered from 0.83 to 0.31. The elevated temperature properties (500 F) are not expected to differ greatly from the room temperature values since the role of the resin has been reduced by the ply orientation. The elevated and room temperature strength of the laminate were confirmed in laboratory tests.

5.2 STEPPED JOINT

The previous boron polyimide box beam employed titanium shim stock in the metal end fittings of the cover skins and spar skins. These shims permitted the fabrication of a stepped joint as the laminate was laid up. Since the permissible thickness of the titanium was controlled by the thickness of the adhesive used to bond shims together as well as the thickness of a ply of composite, the metal in the joint was approximately two-thirds of the laminate thickness. Element tests of the laminate-to-shim bond area showed the metal strength to be controlling the joint strength.

For the graphite polyimide beam, a design decision was made to make the laminate strength control the failure mode of the beam. Therefore, the metal portion of the joint was increased by using a solid titanium part which was stepped by means of chem-milling (see Figure D-4 in Appendix D). Four steps were provided on each side of the titanium part, three for the bond of the outer spanwise layers of composite and one for a double layer of 45-degree material. The two chordwise layers of material were butted against the titanium since the negligible spanwise loads in these layers did not require a shear bond for joint efficiency. The 45-degree layers also were doubled-up because of their low spanwise load. Three-quarter inch

Table 5-1. Room Temperature Material Properties

Property	Material: Boron/Polyimide(1) (5/-12.5/25/-40/45/-45/40/-25/12.5/-5)	Material: Graphite/Polyimide(2) $0_3/\pm45/90)_{2S}$
Modulus (x 10^6 psi)		
Spanwise	16.1	11.9
Chordwise	4.4	5.8
Shear	4.8	2.2
Rigidity (x 10^6 lb/in.)		
Spanwise	0.84	0.75
Chordwise	0.23	0.37
Shear	0.25	0.14
Strength Tension (ksi)		
Spanwise tension	100.0	70.0
Chordwise tension	20.0	32.5
Spanwise compression	70.0	80.0
Chordwise compression	60.0	40.0
Shear	25.0	20.0
Unit Strength (x10^3 lb/in.)		
Spanwise tension	5.25	4.42
Chordwise tension	1.05	2.05
Spanwise compression	3.67	5.05
Chordwise compression	3.15	2.52
Shear	1.31	1.26
Poisson's Ratio	0.83	0.31
Weight		
Density (lb/in.^{3})	0.072	0.058
Areal density (lb/ft^2)	0.542	0.530

Note: 1) Boron/polyimide laminate thickness = 0.053 in.
2) Graphite/polyimide laminate thickness = 0.063 in.

steps were used in accordance with the boron/polyimide design. The performance of this design was established by structural test of two elements of the joint configuration - one at room temperature and one at elevated (500 F) temperature (see Section 6.0). No attempt was made to optimize this joint since the measured performance indicated that the load carrying capability was sufficient.

5.3 SPAN-TO-COVER JOINTS

The selected design for the graphite/polyimide box beam used the same bond for joining the spars and ribs to both the upper and lower cover plates. This design approach differs from the designs developed for epoxy systems and used for the boron/polyimide box in that direct pressure can be applied to the secondary bonds. Since the running shear load in the OV-10A box was 800 pounds per inch, the required shear load was set at 800 pounds per inch.

The design of the shear web and glass/polyimide bonding angles was the same as for one side of the boron polyimide design. Bolts, however, were used at all ends of spar to cover skin bonds in order to reduce the possibility of peel.

6.0 STRUCTURAL ELEMENTS

Structural elements were fabricated and tested to verify selected processes and to establish the performance level of critical sections of the structure. Three types of elements were used - a cover skin stepped-lap bond joint element, a spar web element, and a spar-to-cover element. The first of these elements was selected to verify the integrity of the co-cured graphite/polyimide-to-titanium bond. The second measured the shear capability of the spar in the vicinity of the titanium-to-composite transition. The final element was selected to verify the bond of the glass polyimide spar caps to the cover plates.

6.1 COVER STEPPED LAP JOINT

Two 8.9-inch wide integrally molded/bonded structural elements representing the titanium end fitting-to-composite cover skin joint were prepared in accordance with Figure 6-1. The titanium end fittings were positioned at each end of the test element. They were dimensioned to match the 5.2 mil ply thickness of the graphite/polyimide composite with a total adhesive bond thickness of 2.5 mils, including primer. Actual dimensions after chem-milling the 3/4-in. long steps in the titanium were in good agreement with these dimensions. The attachment hole pattern in the solid titanium section duplicated the hole pattern in the cover plates of the box beam.

The first element was tested in tension according to the following schedule:

1. Room temperature test to 18,000 pounds.

2. 500 F test to 18,000 pounds after a one-half hour soak at temperature.

3. 500 F test to failure after return to zero load from previous test.

The 18,000-pound load applied in both room and elevated temperature loadings represents a load intensity of 2,022 pounds per inch. This is equivalent to a stress of 31.8 ksi in the 0.063-inch thickness skin of the element. Strain gage data showed elastic behavior at both room and elevated temperatures.

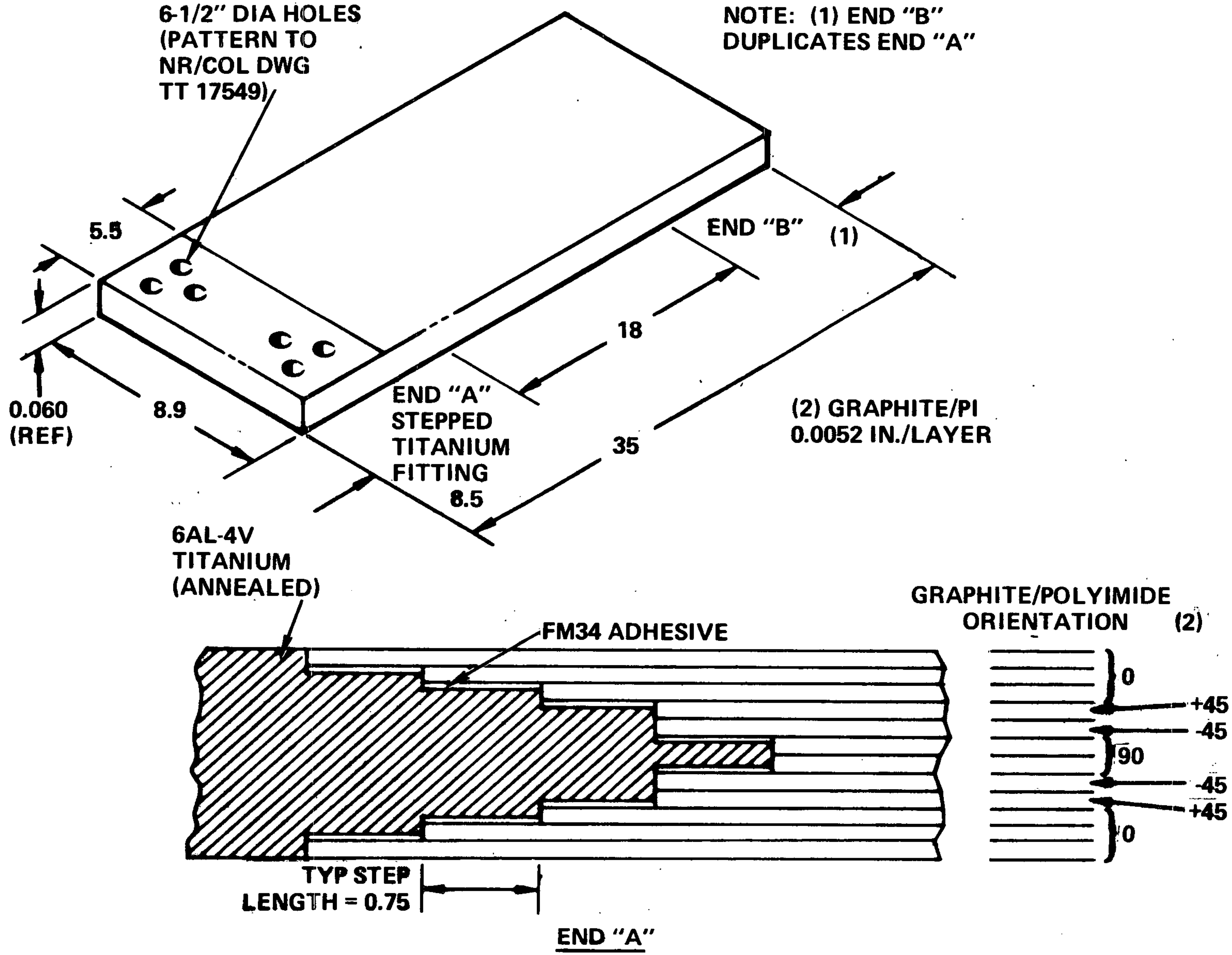


Figure 6-1. Co-Cured Double-Step Lap Structural Element

Panel failure occurred at a total load of 30,400 pounds — a load intensity of 3,420 pounds per inch. The fracture was located at the transition of the first to second steps in the titanium end fitting. The longitudinal stress at failure of 53.7 ksi was approximately 77 percent of the 70 ksi room temperature strength calculated for the laminate from unidirectional test data, ply orientations, and thermal strain.

On first inspection, it appeared that the joint failure was caused by a fracture of the annealed titanium (6 Al-4V) at the transition between the first and second steps. However, a comparison of the extensional rigidities (moduli x thickness) between the longitudinal graphite polyimide composite extending over the step and the 4 to 5-mil thick titanium show the composite to be approximately one order of magnitude stiffer than the metal (0.63 lb/in. versus 0.08 lb/in.). Also, the load in the ± 45 degree composite layers ending at the failure zone were carrying little of the axial load because of their low rigidity. Therefore, the load transferred to the titanium at the first step would be small. Local yielding of the titanium (evident at the break) would cause a major portion of any additional load to transfer to the more rigid composite. Fracture of the panel, therefore, was initiated in the longitudinal composite layer adjacent to the start of the second step in the metal as the result of the concentrated load in this layer. This concentrated load was produced, in part, by the large axial load required to be transferred to the second step in order to force its extension to match that of the composite. This load could only be transferred through the layer of composite next to the titanium. Test data from graphite/epoxy stepped lap specimens of another study (Ref. 7) also have shown similar shear lag effects resulting in failure of the longitudinal layer nearest the titanium adherend. Failure stresses developed in these specimens were 73 percent of the basic laminate strength. Based on this analysis of the failure mode, the observed yielding of the titanium was judged to have occurred as a secondary effect.

The second titanium end fitting-to-cover joint element was tested at room temperature in tension. Failure of this specimen occurred at 33,850 pounds at the same location as the failure of the specimen tested at 500 F — the junction of the first and second step. The load intensity of failure was 3826 pounds per inch, corresponding to a stress level of 59.8 ksi in the composite. This stress is only 85 percent of the 70 ksi R.T. strength developed in specimen tests. Since this stress is greater than the elevated temperature strength, the elevated condition was assumed to be the critical design condition for the box beam.

6.2 SPAR WEB ELEMENT

A structural element representing the spar web configuration, including glass fabric/polyimide laminate close-outs and co-cured titanium-to-composite stepped lap end fitting bonds, was tested under four-point beam loading at room temperature in accordance to the test set up shown in Figure 6-2. Specimen configuration is also shown in the figure.

Failure of the element occurred at a load of 12,400 pounds in the composite adjacent to a titanium end fitting. The ultimate shear load in the composite was 1537 pounds per inch compared to a running shear of 800 pounds per inch required by the OV-10A box beam.

6.3 SPAR-TO-COVER ELEMENT

The spar-to-cover element is defined in Figure 6-3. A dimensional error limited the data obtained on this specimen to processing verification. The specimen was intended to be loaded in compression in a 45-degree direction to the caps. This would produce shear force in the cap and in the bond between the cap and the spar. The dimensions of the specimen would not have permitted development of meaningful data. Therefore, no test was performed.

The dimensions did not produce a square element that would permit the compression loads applied at opposing corners to be reacted by the 45-degree material in the web.

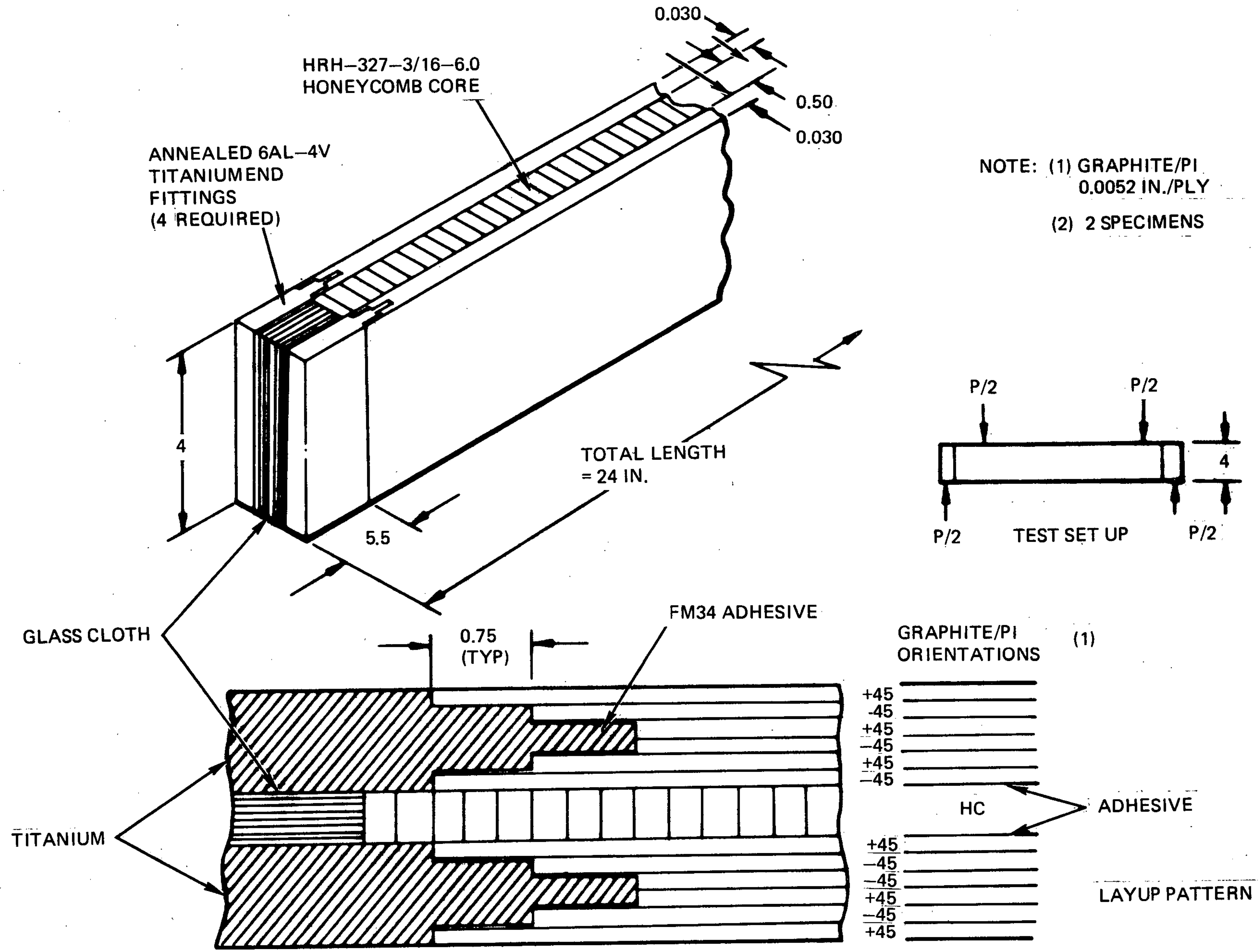


Figure 6-2. Web Element

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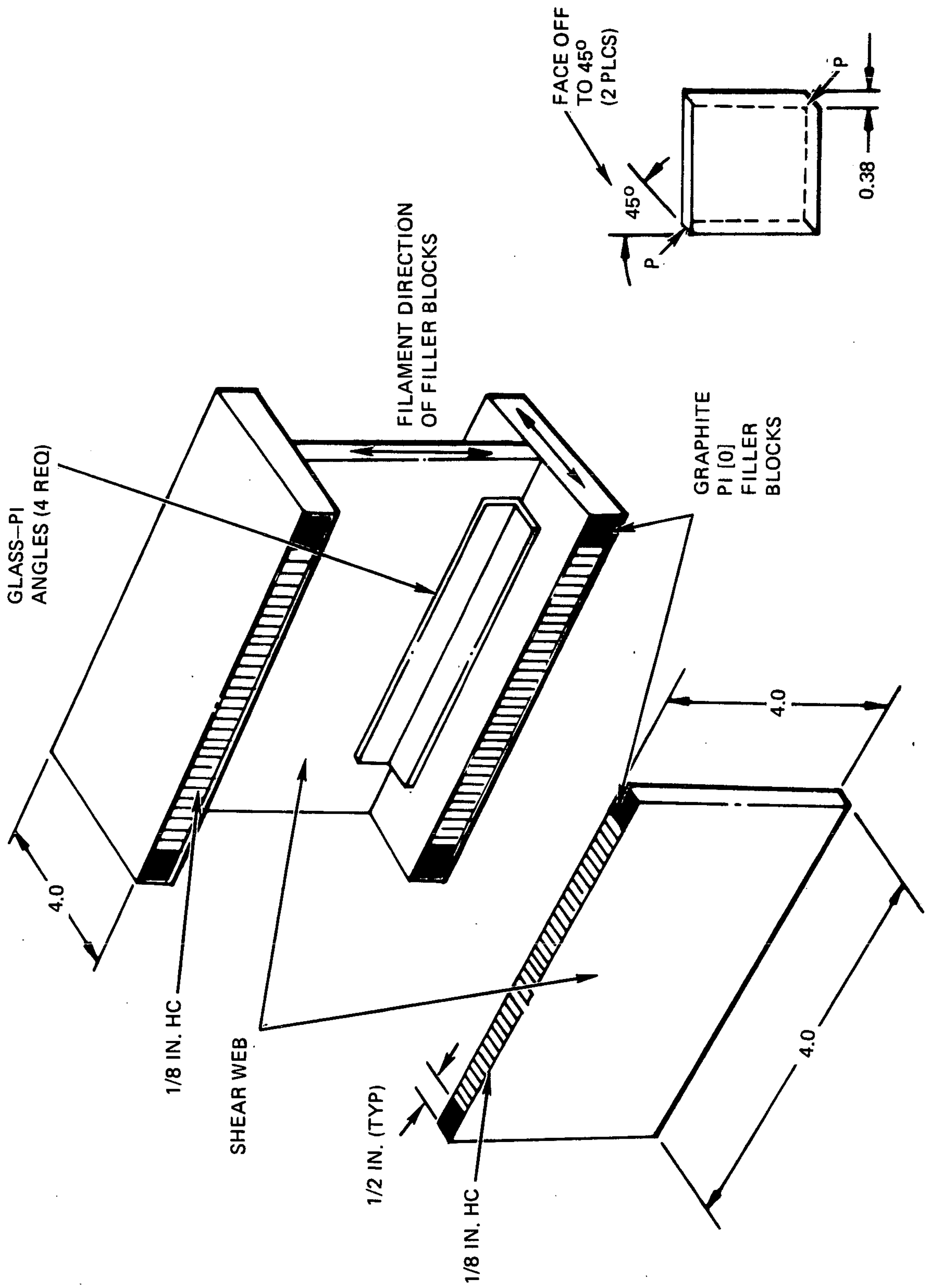


Figure 6-3. Spar-to-Cover Element

7.0 ARTICLE FABRICATION

The G/PI wing box beam article was fabricated per the manufacturing sequence summarized in Table 7-1. This table also contains information highlights on the basic cure and post-cure parameters used, machining operations, the type of process verification coupons employed, and the type of equipment used.

A more detailed process description is contained in Appendix C. The relation of the processes described to the various components, the rib-to-spar subassembly, and the final assembly is discussed in succeeding paragraphs of this section.

7.1 TOOLING

The following special tools were employed, in addition to aluminum lay-up plates:

SK-015652	Rib tool
SK-015654	Lay-up tool for spar face sheets
SK-015655	Application template for spar face sheets
SK-015656	Spar web sub-assembly tool
SK-015657	Lay-up tool for spar angles
SK-015658	Spar bonding tool (angles and caps to web)
SK-015660	Application template, upper cover panel
SK-015661	Bonding tool, upper cover panel
SK-015662	Lay-up tool, upper panel door opening Z-section
SK-015666	Bonding tool, upper panel door

Table 7-1. Graphite Polyimide Manufacturing Process

Part or Assembly	Cure			Post Cure		Machining	Process Verification	Remarks
	Temperature (F)	Pressure	Special Tooling	Temperature (F)	Time (Hours)			
Rib								
Channel fab	350	Vacuum +70 PSI	SK-015652	350 F - 600	4	None	Flexure (0°)	Autoclave ± oven
Cap fab	350	Vacuum +70 PSI	SK-015652	350 F - 600	4	None	Flexure (0°)	Autoclave ± oven
Core/channel/cap bond	350	Vacuum +40 PSI	SK-015652	400 500	4 4	Trim	Lap shear	Autoclave
Spar								
Skin/fitting assembly	350	Vacuum +90 PSI	Al plate	500	18	Trim side and insert location	Lap shear and flexure	Autoclave
Edge inserts	350	Vacuum +90 PSI	Al plate	550	4	Trim to size	None	Autoclave
Doubler (T1)						Cut out		Clean and prime
Core								Splices - cure with bonds
Angles	350	Vacuum +70 PSI	SK-015657	350 F - 600	4	Trim + machine both sides	Flexure (0°)	Autoclave
Molded Inserts								As purchased NR/COL
Caps	350	Vacuum +70 PSI		350 F - 600	4	Trim to size	Flexure (0°)	Autoclave ± oven
Skins/core/molded and edge inserts (subassy A)	350	Vacuum +40 PSI	SK-015658	400 500	3 4		Lap shear and flatwise tension	Autoclave ± oven
Subassy A/caps/angles/doublers	350	Vacuum +40 PSI	SK-015658	400 500 + 1)	4 1)		Lap shear	Autoclave
Cover Panels								
Skin/fitting assy - lower	350	Vacuum +90 PSI	SK-015669	500	18	Trim	Lap shear and flexure	Autoclave
Edge inserts	350	Vacuum +90 PSI	Al plate	550	4	Trim to size	None	Autoclave
Core								Splices - interlock
Skins/core/edge inserts	350	Vacuum +40 PSI	SK-015661	400 500 +	3	None	Lap shear & flatwise tension	Autoclave
Skin/fitting assy - upper	350	Vacuum +90 PSI	SK-015669	500	18	Trim-cut door panels	Lap shear & flexure	Autoclave - store door blanks
Edge inserts	350	Vacuum +90 PSI		550	4	Trim to size	None	Autoclave
Core								Splices - interlock
Door insert and closeout	350	Vacuum +70 PSI	SK-015667	350 F - 600	4	Trim	Flexure (0°)	Autoclave ± oven
Skins/core/edge inserts and bond closeout to panel	350	Vacuum +40 PSI	SK-015661	400 500 +	3 4	Machine door opening to fit angle of closeout and trim to door fit	Lap shear and flatwise	Autoclave - dummy plug
Door								
Upper skin						Cut from lower skin upper panel		
Lower skin						Cut from upper skin upper panel		
Closeout	350	Vacuum +70 PSI	SK-015666	350 F - 600	4	None	Flexure	Autoclave ± oven
Core								Cut to door shape
Bond details	350	Vacuum +40 PSI	SK-015666	400 500 +	3 4	Trim to cover opening	Lap shear and flatwise tension	Autoclave
Assembly								
Rib-spar	350	Vacuum +55 PSI	SK-015672	400 500 + 1)	4 4		Lap shear	Autoclave 2)
Rib-spar to covers	350	Vacuum +55 PSI	SK-015672	400 500 +	2 2	Drill out molded insert	Lap shear	Autoclave 2) ± oven Fill exposed core edges
Installations								
Bearings								
Door								

1) Concurrent with final assembly 2) Autoclave used for heating only; test applies positive pressure

SK-015669	Lay-up tool, upper and lower cover panel face sheets
SK-015671	Bonding tool, spar-to-rib subassembly
SK-015672	Bonding tool, final assembly

The operational features of these tools are in the main straightforward and conventional, with the exceptions discussed below:

The Rib tool, SK-015652, employs two male steel halves for lay-up and cure of the channel facing details. These halves are used in a dowel pin coordinated mode for assembly and bonding of the channel, core, and cap members to each other.

As illustrated in Figure 7-1, the spar-to-rib subassembly bonding tool, SK-105671, confers positive bonding pressure to the vertical rib cap-to-spar web joints by means of air pressure-actuated movable cylindrical plug and rectangular bar members. These members are located over the respective bonding areas of the forward spar. The pressure is translated to the forward bonding areas via interposed in-process control coupons, with succeeding pressure translation by the ribs to the rear spar bonding areas. Additional control coupons are located between the rear spar and the upright rear tool members which are fixed location-wise to the base plate. Connecting tie bars between front and rear upright tool members provide additional tool rigidity.

The final assembly bonding tool, SK-015672, shown in operation in Figure 7-2, functions similarly to the subassembly bonding tool and uses the same base plate. The pressure actuated force members are retained in the H-frame upper member. The exerted pressure is reacted by stout turnbuckle clamps connecting the base plate with the H-frame.

7.2 FABRICATION PROCEDURES

<u>Details</u>

Thin Glass Fabric/Polyimide Details

All thin glass fabric/polyimide details, such as the rib channels, rib caps, spar caps, and spar angles were made from 35-512 prepreg. Lay-up and cure took place per Section 3.4 of the Process Description, Appendix C.

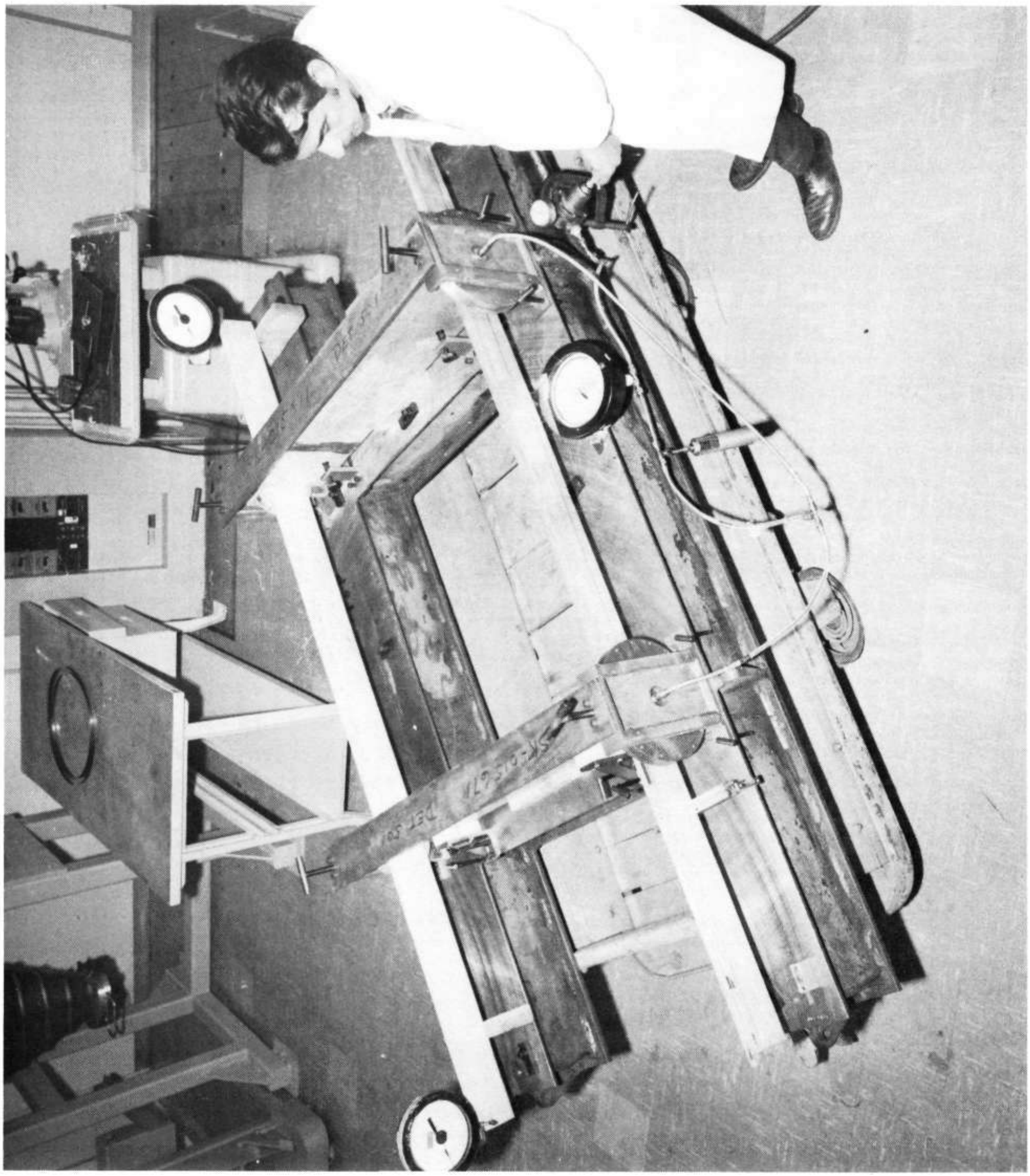


Figure 7-1. Rib-to-Spar Subassembly Bonding

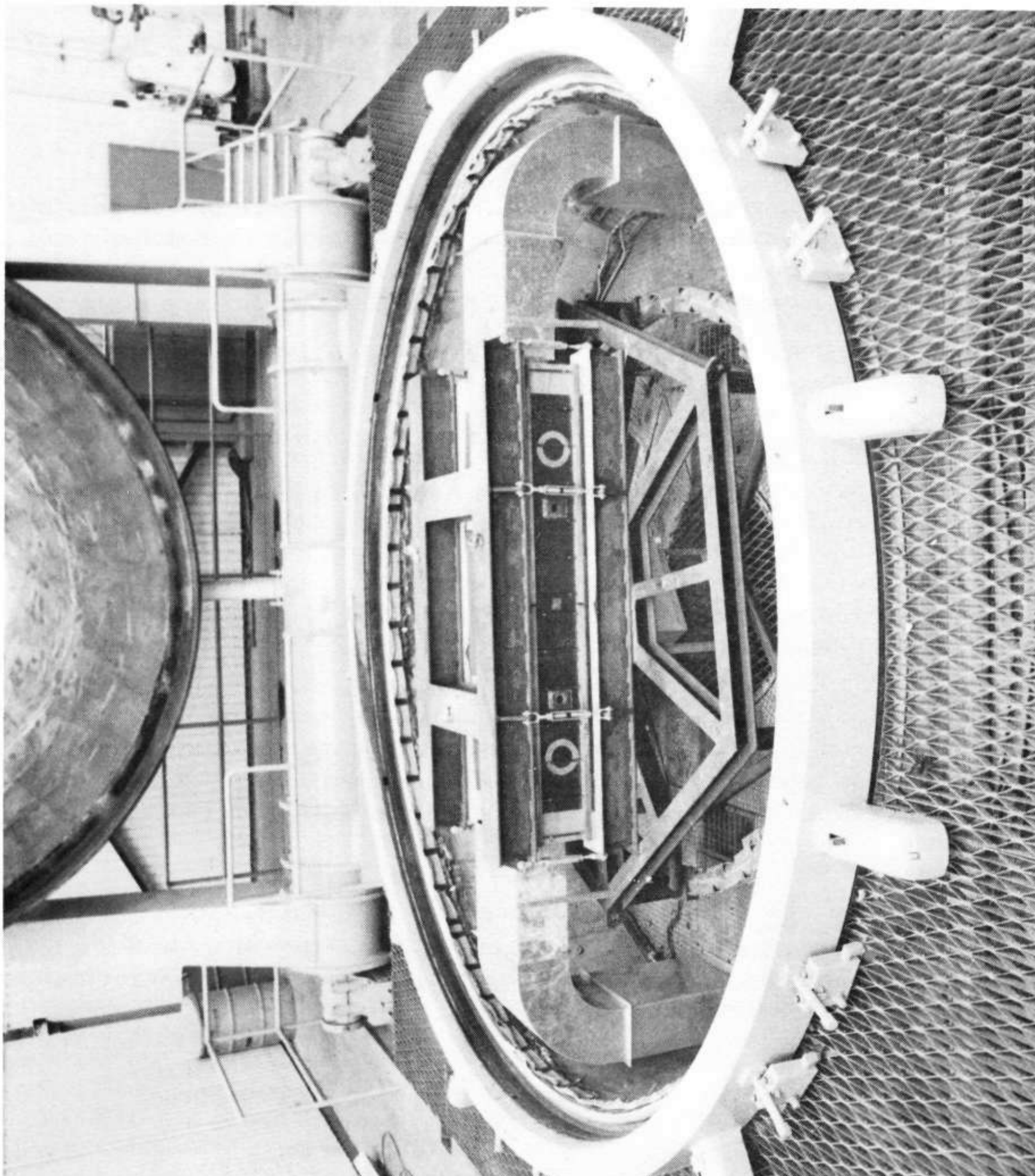


Figure 7-2. Final Assembly Bonding

Thick Glass Fabric/Polyimide Details

35-503 prepreg was used for the following thick glass fabric/polyimide laminate details: Edge close-outs of spars, edge closeouts of top and bottom cover panels, and extension pieces for the bearing inserts. The lay-up processes essentially employed one ply of dry glass fabric after each three prepreg plies with a final two prepreg plies. Lay-up and cure cycle are defined in Section 3.4, Appendix C.

Random Fiber Bearing Inserts

The spar bearing inserts, made from random glass fiber and polyimide resin, were NR/COL furnished parts. They were molded in matched tooling using conventional debulking and curing techniques. Prior to curing, layers of boron/polyimide prepreg were incorporated on the flat surface along each edge of the part.

Titanium End Fittings

Chemical milling of the 6AL4V titanium details for the spar and cover skin end fittings demanded more meticulous processing than is normally required for titanium because of the ±0.001-inch targeted tolerance of each step.

After the titanium sheet stock was sheared and cleaned (MAO 110-024), maskants were applied on both surfaces using Turco Maskant No. 531 and Topcoat No. 550. The maskant was then cut and peeled, exposing the first step area on both sides of the panel. The panel was then mounted on an agitator cathode rod for back and forth movement in the etch bath for uniformity of etch and prevention of undercuts in the step areas. Each step was calculated to prevent excessive removal during the succeeding step-milling operations.

The etch rate was held to 0.0002 inch per minute per surface to avoid channeling. A wetting agent (sodium lauryl sulfate) was added, and the solution temperature was maintained between 100 and 110 F. The formulation of the solution is listed as follows:

Nitric acid	25-30% by volume
Hydrofluoric acid	3-5% by volume
Distilled water	Remainder
Sodium lauryl sulfate	0.0005 to 0.0017% by weight

Honeycomb Core

Processing of the HRH-327-3/16 polyimide honeycomb core blanks used in the rib, spar, cover panel, and door components is described in Paragraph 3.6.

In the overlap/interlock splicing technique used for the spars, 1 inch of core was overlapped while the cells were offset on a metal platen. A wood plank 2 by 4 inches was placed over the overlap area, and the pieces were compressed into an interlock joint by pounding with a mallet.

Graphite/Polyimide Composite Details

The spar web facings and the facings for the upper and lower cover panels were laid up and integrally cured and bonded to their respective titanium end fittings per Sections 3.4 and 3.6. The spar skins were laid up double width, and sectioned after post cure to attain the individual facings. Door skin blanks were obtained by appropriate cut-outs from the upper cover panel facings.

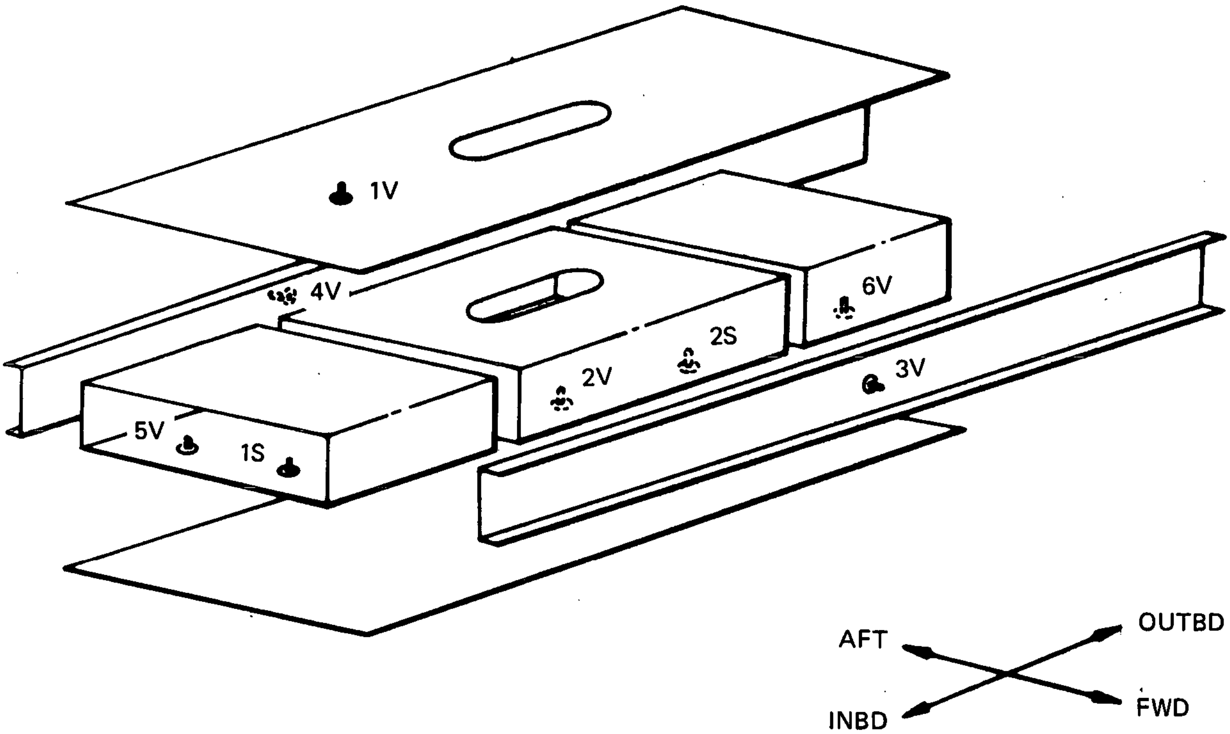
Component Fabrication

Adhesive assembly of the details for the ribs, spars, upper and lower cover panels, and the door was accomplished with the aid of their respective tools after prefit as described in Section 3.6.

Spar-to-Rib Subassembly

The bonding operation for this subassembly was performed per Section 3.6. Rather elaborate provisions for vacuum line (operational and static) and thermocouple monitoring were made, Figure 7-3 and 7-4, in dummy (pre-fit) runs and in the actual bonding operation. This permitted establishment of proper operational settings prior to commitment, and assurance that bond line temperatures at all locations were within target ranges during cure.

A bag leak with complete vacuum loss developed in the first pre-fit run approximately 1-1/2 hours into the heat-up cycle, due to opening of one of the zinc chromate vacuum putty sealed joints. A knife slit also was discovered in post-run inspection. Very low RT tensile shear strength results were obtained on the control coupons accompanying this run, 200 to 800 psi.



V = VACUUM
S = STATIC

Figure 7-3. Spar-to-Rib Subassembly Vacuum and Static Line Locations

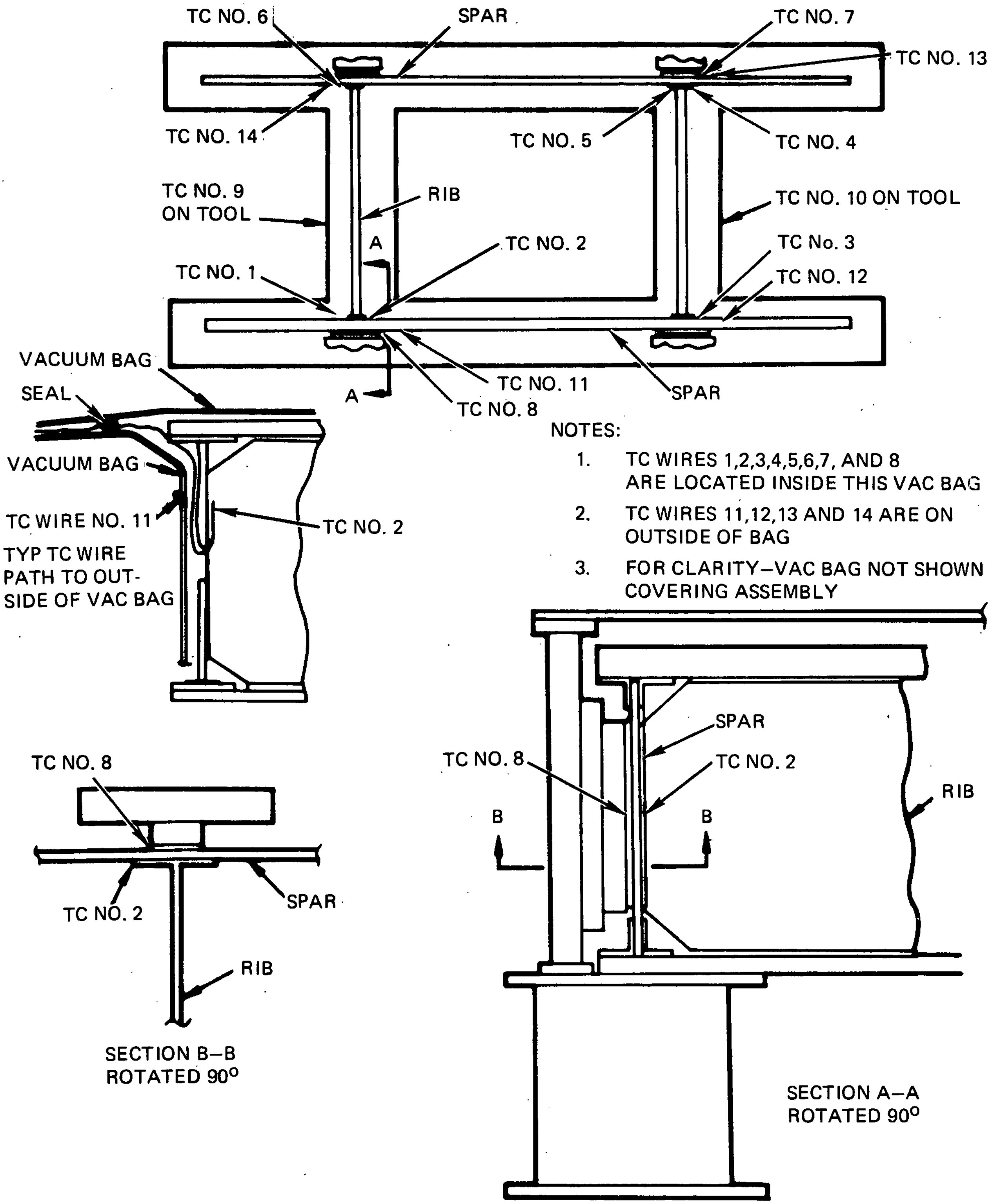


Figure 7-4. Thermocouple Locations Spar-to-Rib Subassembly

A second dummy run, with a more secure support arrangement for the bag joints was satisfactory except for low readings (20 - 22 in. Hg) on the static vacuum gages of the autoclave console. Checks against portable gages at bag port locations produced readings of 27+ and 28.5 in. Hg, indicating leakages in the console vacuum read-out system. Since no timely repair was possible, static vacuum gage readings during subassembly bonding and the subsequent final assembly trial and actual bonding runs were used only as guides.

The pre-fit checks indicated that one ply of FM-34, 0.135 psf adhesive would provide proper glue lines for all rib-to-spar joints; one ply was therefore used throughout in the actual bonding operation.

Assembly Bonding

The rib-spar subassembly was bonded to both upper and lower cover panels in a single operation per Section 3.6. The location of the thermocouples used to monitor the trial and the actual run is shown in Figure 7-5. Bag joint leaks were encountered in the pre-fit run, leading to the decision to use permanent DC92-018 bonds for all bag joints in the actual run. The dummy run proved that the tool had applied satisfactory pressure to the part. Verifilm FM 641 thicknesses were as follows:

Location	Maximum (Inch)	Minimum (Inch)
Aft spar bottom	0.019	.004
Forward spar bottom	.020	.002
Inboard rib bottom	.023	.0035
Outboard rib bottom	.036	.004
Aft spar top	.017	.0035
Forward spar top	.021	.003
Inboard rib top	.017	.0035
Outboard rib top	.019	.003

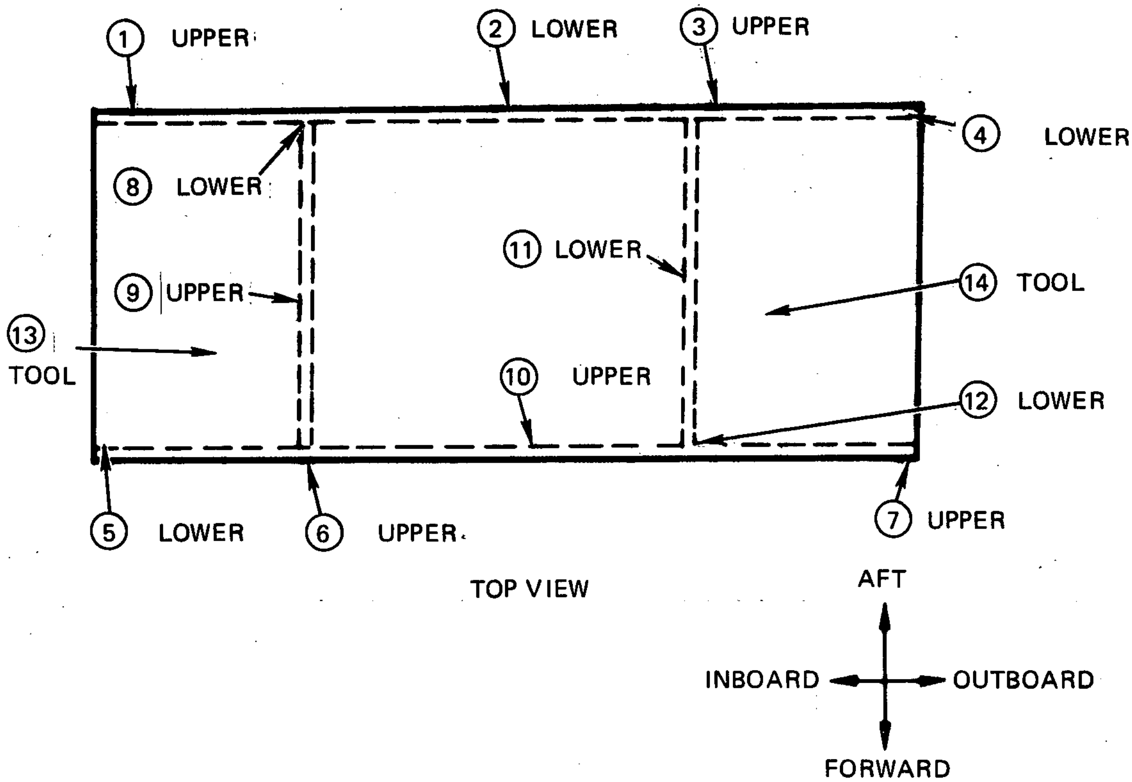


Figure 7-5. Thermocouple Locations, Final Assembly Bonding

Based on these measurements, two layers of FM-34, 0.135 psf adhesive were used throughout, except for a 15-inch long section on the top of the inboard rib employing three layers.

YM-25 compound was applied to all core edges still exposed after the bonding operation. The post cure cycle used includes hold steps 200 F and 300 F to permit adequate release of volatiles from the edge fill compound and its simultaneous cure with post cure of the assembly.

Door Installation

Fasteners were installed and the door mated with the cover-spar-rib assembly per Drawing MT-100017, Figure D-7 in Appendix D. A photograph of the completed test article is shown in Figure 7-6.

Repairs

Discrepancies noted during various fabrication steps were repaired as follows:

700-86-1139A

Figure 7-6. Completed Graphite/Polyimide Box Beam

Rib Caps

Rib cap delaminations were encountered in the four locations facing the spars on disassembling after the first subassembly orientation trial run. It was evident that sticking of the presumably "nonstick" Verifilm 641 impression check film was the responsible factor. The four rib caps were rebonded with FM-34. The Verifilm material was sandwiched between 0.001-inch thick Kapton film in all subsequent impression check runs.

Re-delamination of the aft cap of the inboard rib occurred on drilling the holes for the assembly aid fasteners due to lack of providing fully clamped support during this operation. The cap was rebonded with FM-34.

Further problems were encountered with local rib delaminations in the first and second dummy bonding test runs. The cause of these delaminations was traced to mechanical locking of the Verifilm around the assembly aid bolts, preventing trouble-free disassembly. Repair was effected by rebond with FM-34 and local injection with BR-34 and insertion of FM-34, respectively. Teflon washers were used in cut-outs of the Verifilm in final assembly dummy runs to prevent a similar occurrence.

Spar Angle

The legs of the initial spar angles produced were not at 90 degrees to one another. They were repaired by post-forming. For the remainder, the lay-up tool was corrected to accommodate the spring-back observed.

Spar Cap

A local delamination in one of the spar caps, noted after the final assembly dummy run, was repaired by injection of BR-34 primer and insertion of FM-34 adhesive.

Upper Cover Face Sheet

BR-34 injection and insertion of FM-34 also was used for repair of 3/4-inch delamination in the top upper cover face sheet. Cure was effected simultaneously with the cover panel component bonding operation.

8.0 QUALITY ASSURANCE

Quality assurance measures entailed receiving inspection tests on productive materials against purchase orders or applicable material specification requirements; recheck as applicable from storage period expiration considerations; tests on control coupons accompanying the various processes (fabricated under the same bag under identical conditions); and appropriate dimensional checks.

8.1 INCOMING MATERIALS

The major mechanical properties acceptance criteria for the productive materials used in the box beam members are shown below. Reference was also made to the Gemon L/Modmor II Procurement Specification, Appendix A, and an existing material specification for Polyimide Adhesive, MB0120-062, for additional requirements on these materials.

Acceptance Criteria, Productive Materials

Modmor II/Gemon L	Min. Average	Min. Indiv. Value
R.T. Flex. Str., ksi	175	160
500 F Flex. Str., ksi	115	105
R.T. Flex. Mod., msi	15.0	14.0
500 F Flex Mod., msi	13.0	12.0
R. T. Short Beam Shear Str., ksi	11.0	10.0
500 F Short Beam Shear Str., ksi	6.0	5.5

Glass Fabric/PI, 35-512 and 35-503

Flexural Strength at 500 F - - - - - - - - 45 ksi min.
Flexural Mod. at 500 F - - - - - - - - 2.1 msi min.

FM-34 Bonds

1. Specimen configuration Ti-to-Ti adherends, 1/2-inch overlap, single lap for tensile shear. Two layers of adhesive are to be used for FM-34, 0.03 psf.

   0.020 Al facings to 3/16-0.002 or 0.003 Al core for flatwise tension.

2. Acceptance criteria.

500 F tensile shear strength	1200 psi
R.T. flatwise tens. strength	500 psi

Glass Fabric/Polyimide Prepreg

All material used exceeded stipulated requirements by comfortable margins.

Gemon L/Modmor II Prepreg

Receiving inspection testing on Modmor II/Gemon L prepreg ordered for fabrication of the box beam indicated substantial non-conformance with the requirements of the procurement specification. In the primary parameter, fiber weight per unit area, the specification calls for 12.6 ± 1.3 gram per square foot on individual measurements and 12.6 ± 1.0 gram per square foot on the average of two or more specimens. Individual test values showed that in tests on 34 sheets, 19 were out of specification on individual values and 17 on averages. To prevent untenable slippage in the schedule it was decided to accept the material despite the obvious discrepancies in fiber weight. Deviations from this parameter were reflected in scale-down of design expectations.

FM-34 Polyimide Adhesive

All productive material used conformed to requirements except for early receiving inspection results in which questionable procedures were used in fabrication of the specimens. Discrepancies were lack of vacuum (the importance of this parameter had not been recognized at that time) and use of an aluminum assembly fixture with fixed pins which did not permit proper bondline conditions for the titanium specimens due to differential thermal expansion. Later retesting revealed all material used substantially met specification requirements.

8.2 IN-PROCESS CONTROL

A process verification plan was set up, calling for "under the same bag" fabrication of control coupons with each laminating or adhesive bonding operation and subsequent testing of these specimens to the criteria of the Paragraph 4.1 of the Process Description, Appendix C. The plan logic underlying Table 7-2 employs flexure specimens for monitoring of laminating operations, (glass fabric and graphite prepreg respectively), lap shear specimens for secondary continuous surface as well as co-cured bonds, and flatwise tension specimens for honeycomb core bonding. Repair operations were similarly monitored.

The results on control coupons accompanying the major operations are summarized in Table 8-1. It may be seen that in many cases, actual tests performed exceeded those required by the basic plan.

No problem was ever encountered in meeting the stipulated values for glass fabric/polyimide laminates, nor for honeycomb bonds.

The requirement of 115 ksi 500 F flexural strength was not met by the undirectional graphite composite control coupons for the spar forward face sheets, nor either of the lower cover panel face sheets. A partial explanation for failure to meet this requirement is the excessive thickness of these control coupons. Although the ply thickness of the details was close to the 5.2-mil target, ply thickness of the control coupons, — though produced under the same bag — was ~5.9 mils. The laminates were accepted since (1) values normalized by a 5.9/5.2 ratio would indicate that the actual load carrying capacity was still close to expectations and, (2) test values on trim from titanium end fitting-to-composite laminate areas were judged to exceed by large margins the stress levels the composite would see in the structural test.

The poor results on specimens accompanying the various co-cured end fitting bonding/composite laminating operations are ascribable to two causes: (1) Pinned fixturing on aluminum jigs which produced undue stresses on bond lines and (2) usage of single in lieu of the stipulated two layers of the extremely thin 0.03 psf FM-34 adhesive. Tests on trim from these panels indicate that the bond strength obtained in the facing details substantially surpasses design and structural test load intensity requirements. These requirements are 2730 pound/inch for cover panel facings and 600 pound/inch for spar facings.

Table 8-1. Control Coupon Data

	Glass Fabric/Polyimide		Gemon L/ Modmor II	Sandwich	Titan. -to-Titan.		Trim, Step Lap	
	Flex Str KSI	Flex Mod MSI	Flex Str KSI	Flatwise Tension KSI	Lap Shear Strength PSI		Shear, Failure Load Intensity, LB/IN	
	500 F	500 F	500 F	500 F	RT	500 F	RT	500 F
Target Value, Minimum	45*	2.1	115*	500*	2500	1200*		
Rib No. 1								
Channels and caps	62.2	2.4						
Component assembly					2540 2711	1380 1798		
Rib No. 2								
Channels and caps	57.1	2.1						
Component assembly					2620	1400		
Spars								
Forward face sheets			102			793 1)	2 10	1059
Aft face sheets			142			763 1)	2310	1506
Angles and caps, forward spar	67.5	2.48						
Angles and caps, rear spar	57.3	2.68						
Subassembly bond				918		1195		
Cap bond						1725		
Lower Cover Panel								
Lower face sheet			97.8			1075	3550	
Upper face sheet			110			835 1)	3610	
Assembly bond				1049		1198		
Upper Cover Panel								
Lower face sheet			117.5			843 1)	3540	
Upper face sheet			123.5			603 1)	3100	
Door insert and close-out	75.7		1					
Assembly bond				657		1378		
Door								
Z-section close-out	75.7							
Assembly bond				2)		2)		
Spar-Rib Subassembly					3194	1358		
Final Assembly					2473	1186		

*Required test
1) Improperly prepared specimens
2) Process control coupons inadvertently omitted

8.3 DIMENSIONS

The following comments are considered of interest with respect to dimensional measurements:

Spar and cover panel components were planned to be fabricated "net". Spar panels produced on aluminum tooling generally were ~1/8 inch longer than intended, cover panel facing half-length varies from 41.694 inches to 42.189 inches versus the intended 42.00 inches. This produced a spar overhang. No attempt at final common trim was made, since the non-match condition would not interfere with fixturing for the structural test.

Surface flatness of the lower panel was within 0.018 inches; total assembly thickness varied from 11.910 inches to 11.930 inches.

An intermediate surface table flatness reading of the rib-to-spar sub-assembly produced the results shown below:

	Top of Assembly*	Bottom of Assembly
Aft spar - center	-.009 to +.007	-.009 to +.015
- edge of flanges	-.009 to +.009	-.031 to +.001
Forward spar - center	-.012 to +.008	-.009 to +.005
	-.022 to +.001	-.029 to +.017
Inboard rib - center	+.002 to +.006	-.015 to .000
- edge of flanges	-.008 to +.005	-.032 to +.007
Outboard rib - center	+.007 to +.010	-.014 to -.001
	-.013 to +.018	-.024 to +.002

*Inches from reference dimension.

It was feared that the subassembly would require machining prior to bonding the covers. However, an inspection of the part revealed most of the tolerance problems occurred at the edges of the caps flanges. Light finger pressure on the flanges appeared sufficient to pull up the mismatch.

The dummy run established that satisfactory fit-up would be achieved under vaccum and 55 psi applied pressure to the cover joints.

9.0 DOCUMENTATION

9.1 INCOMING MATERIALS

The quality of productive incoming materials was documented by vendor's certifications and by NR/SD test reports, Form 963-C-1, Rev. 9-69. A typical report is shown in Appendix E.

9.2 FABRICATION

The design of all tooling and shop aids used on the program was recorded on controlled (SK) drawings. Changes were recorded by E. O.

Supplementing the process description, Appendix C, detailed manufacturing procedures were prepared for each operation, as exemplified in Appendix F. Documentary control was maintained by release of these procedures in the following SK drawings:

SK 015653	Manufacturing Procedure for Rib Fabrication
SK 015659	Manufacturing Procedure for Spar Fabrication
SK 015663	Manufacturing Procedure for Lower Panel
SK 015664	Manufacturing Procedure for Upper Panel
SK 015665	Bagging Procedure for Gemon L/Modmor II Face Sheets
SK 015667	Manufacturing Procedure for Door Fabrication
SK 015668	Manufacturing Procedure for Box Beam Sub-Assembly (Spars to Ribs)
SK 015673	Manufacturing Procedure for Final Assembly

These procedures, as applicable, are called out in the general notes of the pertinent component drawings.

Additional fabrication documentation is contained in Laboratory Notebooks N-10452, N-11240, and N-11910.

Other records are cure record sheets, Form 2972-7, New 12-66 and Temperature Charts.

9.3 DIMENSIONS

Dimensional information is generally recorded in the Laboratory Notebooks.

10.0 GRAPHITE/POLYIMIDE WING BOX BEAM TEST

Simulated OV-10A center wing sections previously have been fabricated from composites and tested to loads scaled from a critical design wing loading condition. Reference 3 discusses the fabrication and room temperature testing of a boron epoxy box beam. Reference 4 describes the fabrication and testing of a similar article fabricated from boron polyimide and tested at +650 F. The elevated temperature was imposed to demonstrate the structural integrity of the polyimide assembly when subjected to external loads in a thermal environment. In both cases, the primary objective was the demonstration of fabrication techniques; test loads were scaled so as not to exceed predicted material allowables under test conditions.

The subject graphite polyimide box beam was tested in a manner similar to the boron polyimide beam. The design load condition was scaled so that the applied ultimate loads resulted in critical internal loads equal to predicted failure values measured in element tests. Ultimate testing was performed at +500 F.

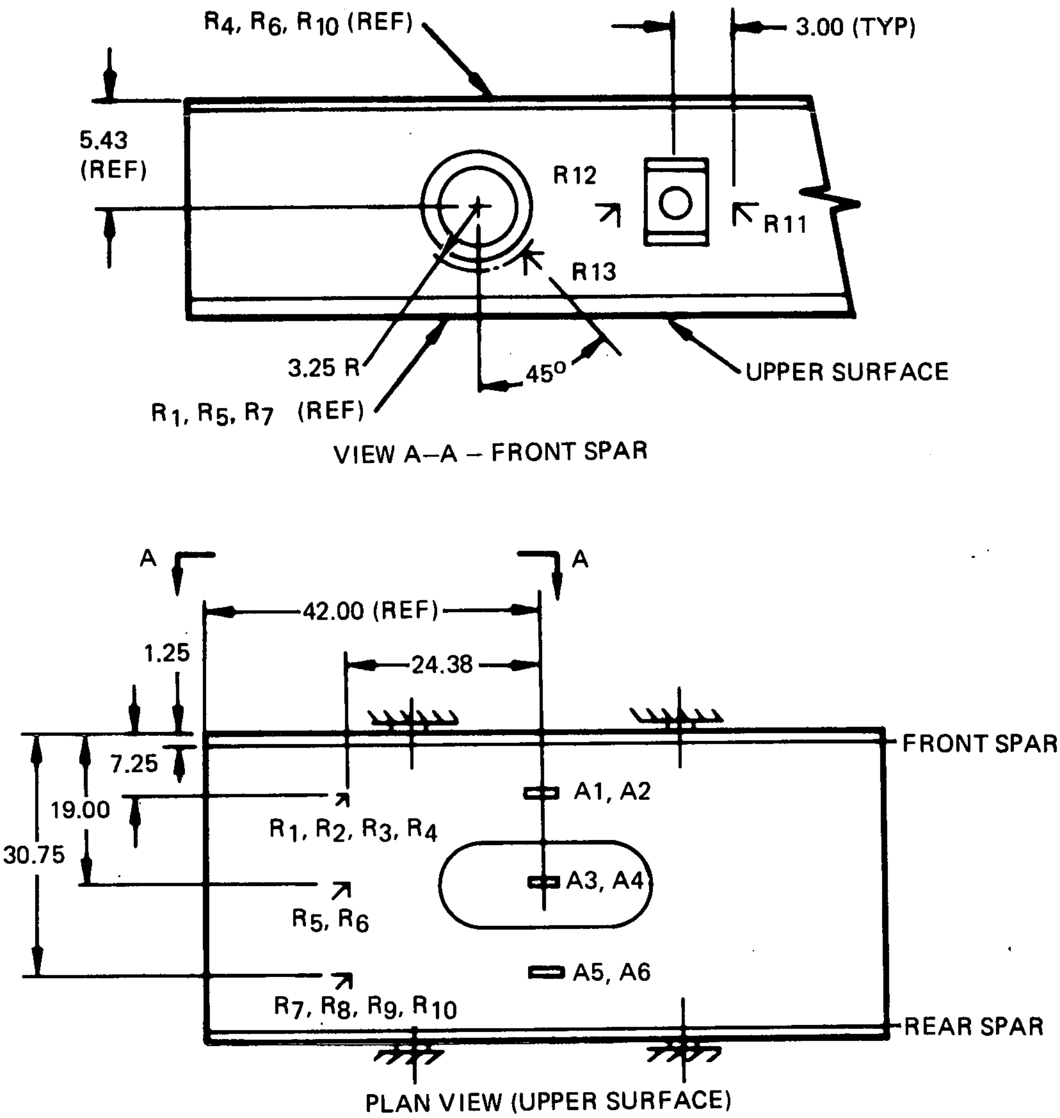
10.1 TEST REQUIREMENTS

Instrumentation

High temperature strain gages were installed as shown in Figure 10-1. Temperature compensation was provided using dummy gages as required for each temperature regime. A total of 16 deflection transducers were installed as indicated in Figure 10-2. Specimen and support deflections were transmitted to the transducers using materials possessing low thermal expansion characteristics (nichrome wire) to minimize errors resulting from the elevated temperature. Twenty thermocouples for monitoring and readout were installed as shown in Figure 10-3. Additional thermocouples (three per reflector bank) were installed as indicated in Figure 10-4 to measure temperatures as required for control of the heater lamps.

Fixtures

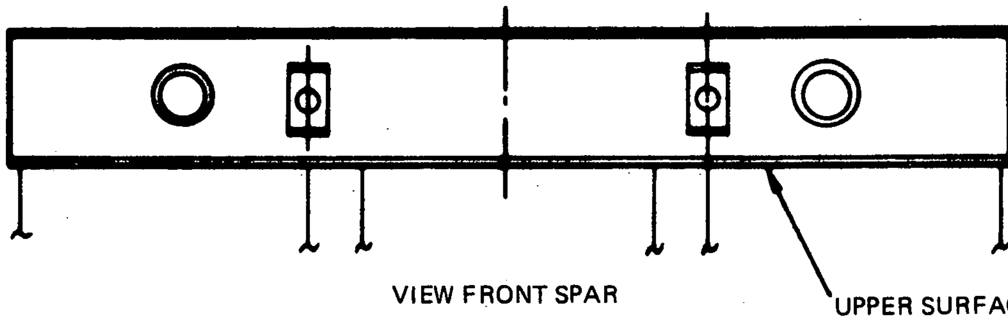
Test fixtures were defined in drawing TT-17542, Test Set-Wing Non-Metallic, Center Box, Assembly of (Test Specimen). The fixtures and test set-up are further described in Reference 4. Thermal insulation of all test fixture-to-specimen attach angles were protected by means of asbestos cloth or similar material to minimize heating of these members during the elevated temperature testing. Figure 10-4 shows the areas to be insulated



NOTE:

1. ROSETTE GAGES ARE DESIGNATED BY "R" AND AXIAL GAGES BY "A"
2. ROSETTE NUMBER SEQUENCE – UPPER TO LOWER SKINS. R_2, R_3, R_8 AND R_9 ARE ON INSIDE SKINS

Figure 10-1. Strain Gage Installation



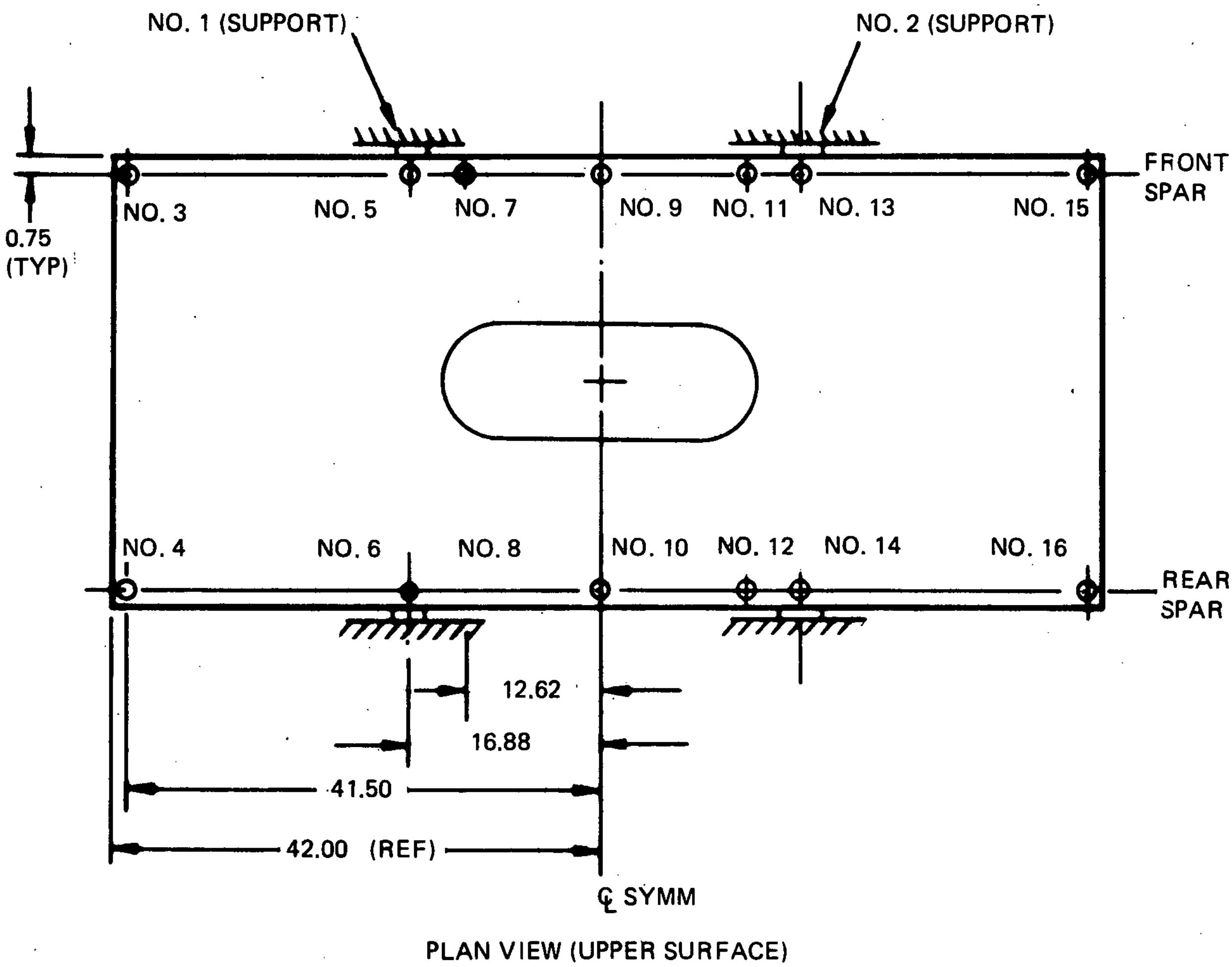
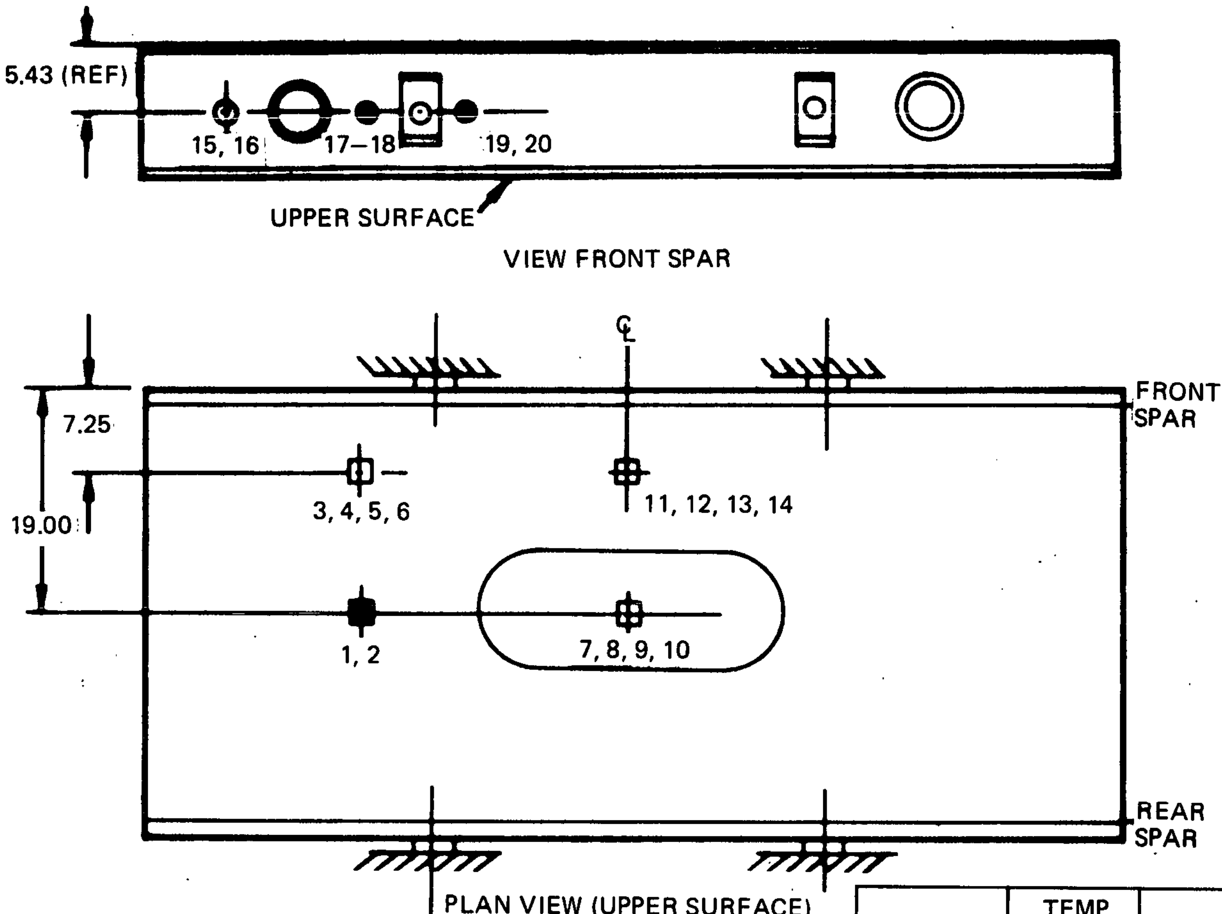


Figure 10-2. Deflection Gage Location

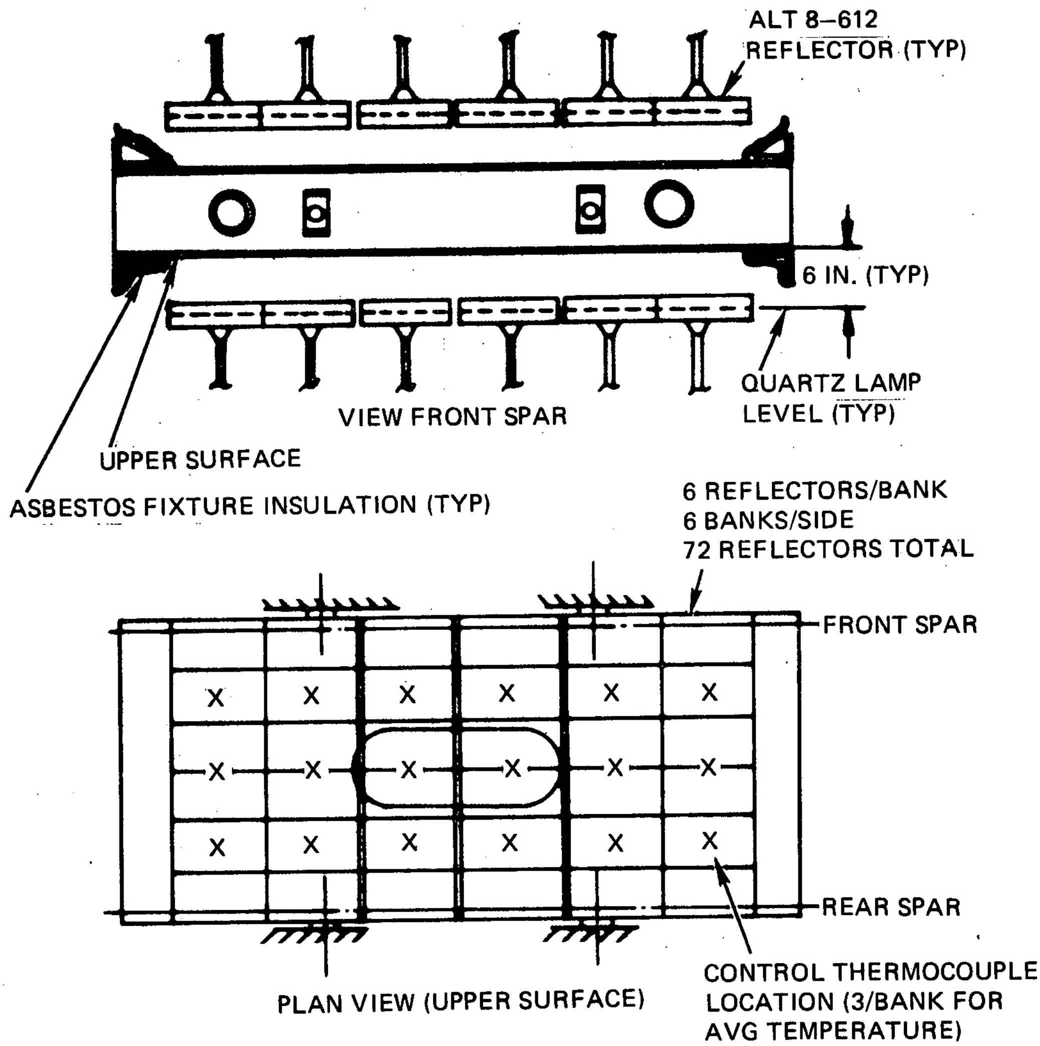


THERMO-COUPLE NO.	TEMP 0 LOAD 5 MIN@ 500 °F	TEMP 70% LOAD
1	478	476
2	476	478
3	471	468
4	250	285
5	304	334
6	456	447
7	491	495
8	405	444
9	378	430
10	491	486
11	484	478
12	344	406
13	385	431
14	504	510
15	110	102
16	112	168
17	118	158
18	128	150
19	121	206
20	117	197

NOTE:
- □ LOCATED INSIDE AND OUTSIDE OF BOTH SKINS
- ■ OUTER SURFACE ONLY OF UPPER AND LOWER SKINS
- ○ INSIDE AND OUTSIDE SURFACE OF FRONT SPAR ONLY
- ● OUTSIDE SURFACE ONLY OF FRONT AND REAR SPARS

THERMOCOUPLE NUMBER SEQUENCE – UPPER TO LOWER SKINS AND FRONT TO REAR SPAR

Figure 10-3. Thermocouple Installation, Graphite Polyimide Box Beam



NOTE: X INDICATES LOCATION OF CONTROL THERMOCOUPLES ––

A TOTAL OF 192 1000 T3CL QUARTZ LAMPS IS USED WITH
72 ALT 8–612 REFLECTOR UNITS

Figure 10-4. Heating Arrangement, Graphite Polyimide Box Beam and describes the general arrangement of heating elements required to achieve the test temperature profile. Figure 10-5 shows the specimen in the inverted test position (compression surface down) and the range of loads for the hydraulic jacks used in the test.

The external load test setup consisted of steel reaction fixtures at 16.875 inches from box center line at both the front and rear spar (total of four spherical bearing reaction points). The shear, moment, and torque were applied by hydraulic struts through universal clevises whiffle tree linkage as shown on Figures 10-5 through 10-7. Pressure was supplied to the hydraulic struts by a hydro-pump and the loads were proportioned by an Edison machine. Loads were monitored at strut attach points by calibrated load links. A master deadman switch was used on this test to instantaneously dump both the hydraulic and electrical input if failure or malfunction occurred. Jig fixture and linkage dead weight were counterbalanced by a hydraulic strut on a system independent of the master dump system.

Conditions

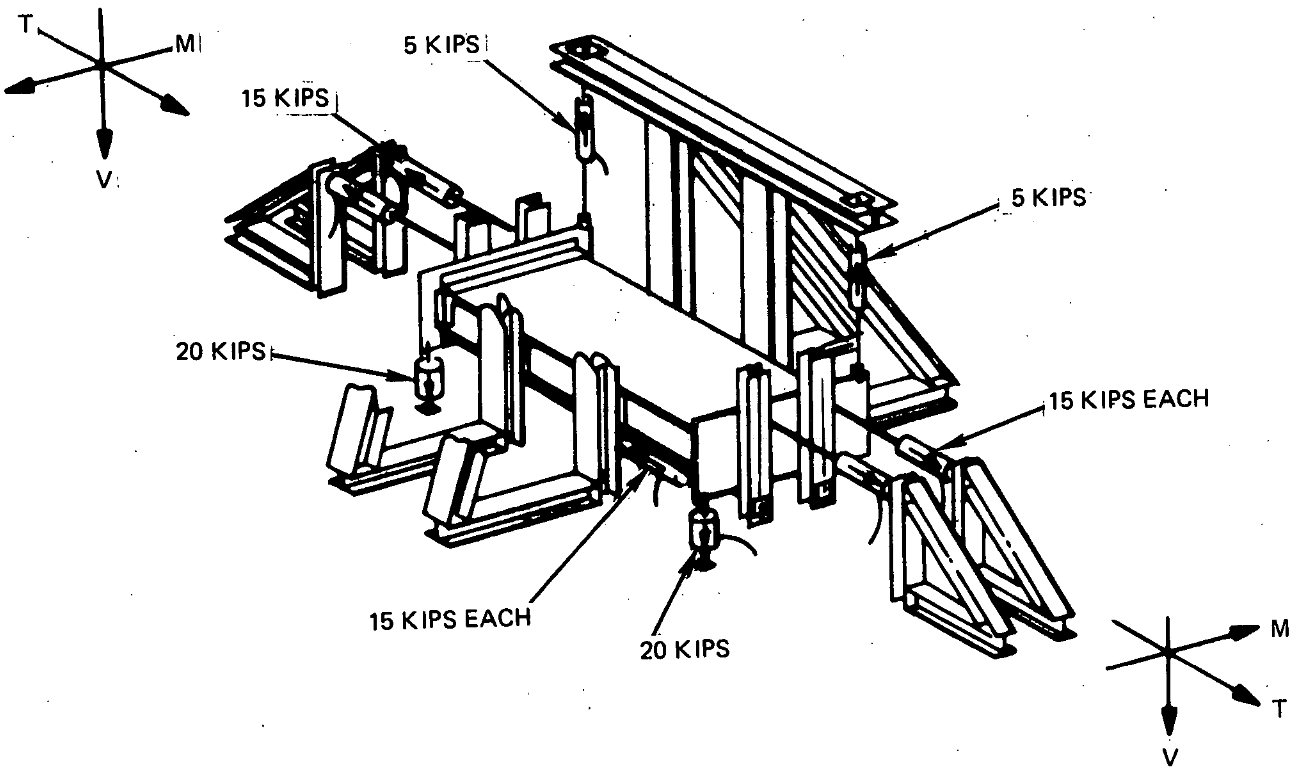
The design ultimate loads were established by scaling OV-10A center wing box loads to provide a match of internal loads with the allowables of the cover skin laminates. These allowables were defined by reducing tensile test data from 8.9-inch wide structural elements by 20 percent to account for quality differences between laboratory specimens and manufactured parts. Based on this reduction, the allowable skin unit load and stress of the skins were set at 2736 pounds per inch and 43 ksi, respectively. (see Paragraph 6.1 for measured cover strengths).

The calculated OV-10A maximum skin load resulting from combined bending moment, shear and torsion loads of 3,150,000 inch-pound, 21,250 pound and 660,000 inch-pound, respectively, was 3925 pound/inch. The preliminary applied ultimate loads for the graphite polyimide test beam of this program were, therefore, scaled to be:

$$M = \frac{2736}{3925} \times 3,150,000 = 2,190,000 \text{ in.-lb}$$

$$T = \frac{2736}{3925} \times 660,000 = 460,000 \text{ in.-lb}$$

$$V = \frac{2736}{3925} \times 21,250 = 14,800 \text{ lb}$$



DESIGN ULTIMATE LOADS:
M = 2,190,000 INCH POUNDS
T = 460,000 INCH POUNDS
V = 14,800 POUNDS

NOTE:
1. STRUT LOADS SHOWN ARE CONSERVATIVE MAXIMUM CAPABILITIES REQUIRED TO ACHIEVE ULTIMATE LOADING
2. BOX BEAM SHOWN IN INVERTED TEST POSITION (UPPER SURFACE FACING DOWN)

Figure 10-5. Structural Test Set-up, Graphite Polyimide Box Beam

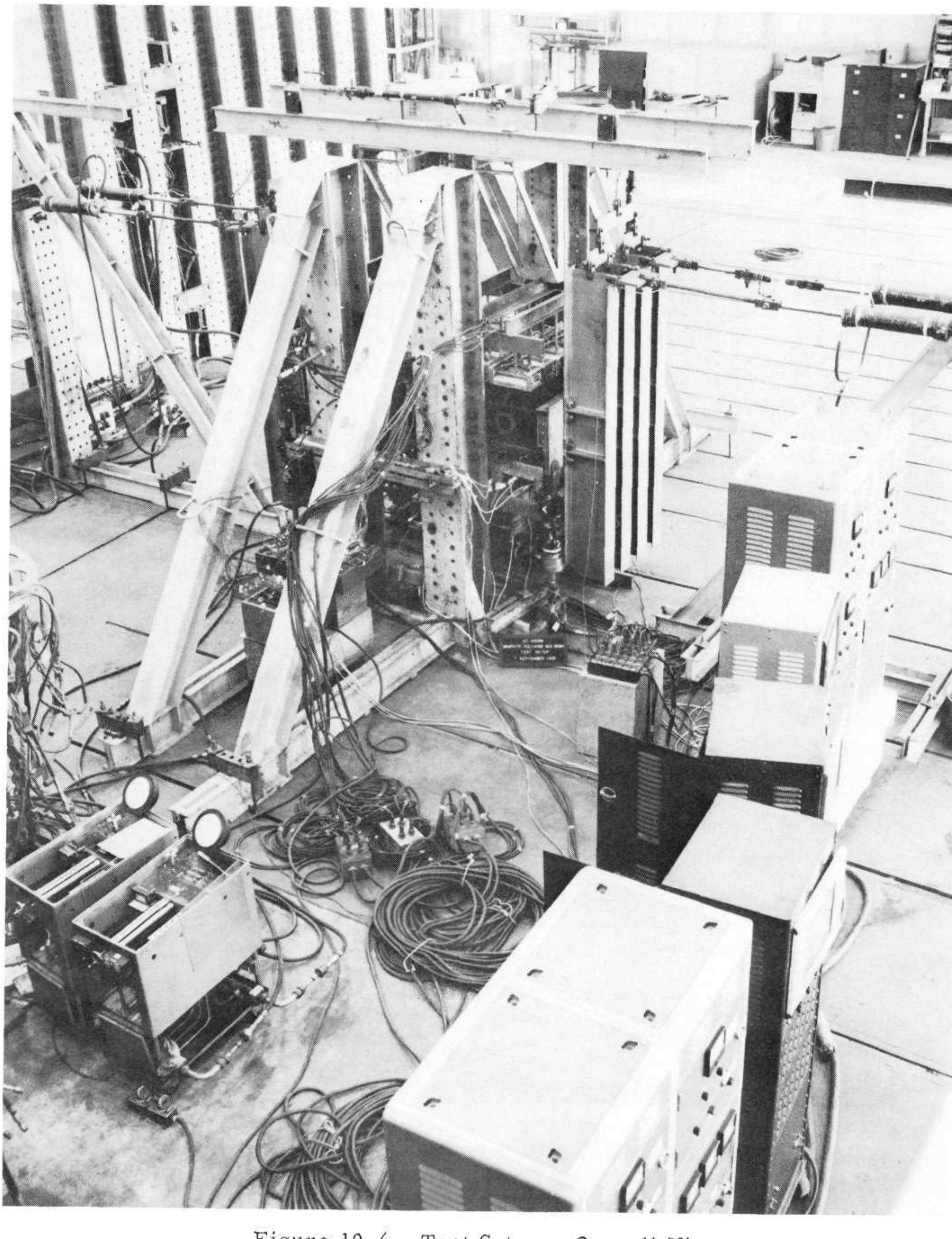


Figure 10-6. Test Set-up, Overall View

Space Division
North American Rockwell

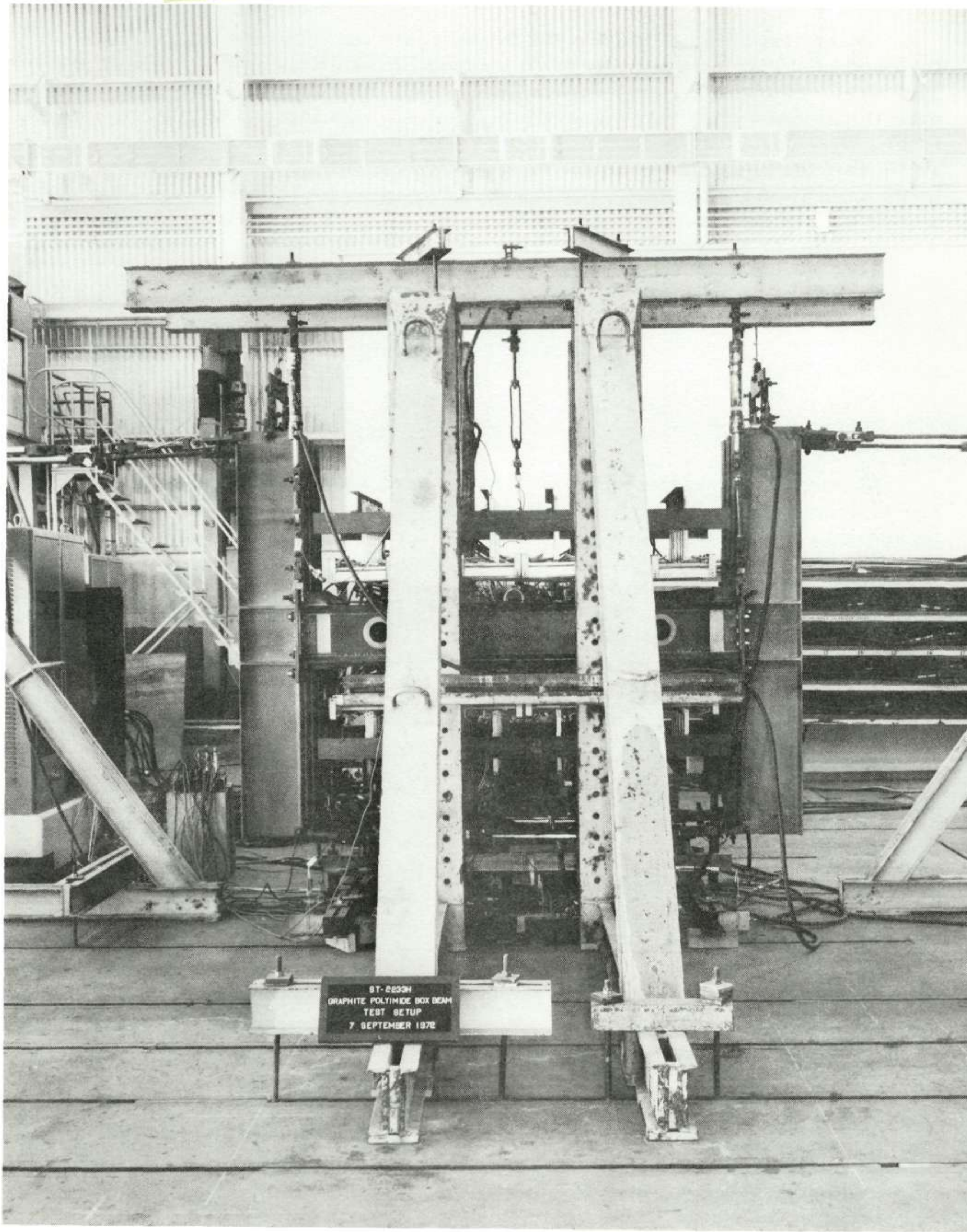


Figure 10-7. Test Set-up, Side View

After test setup of the box beam and room temperature trial runs of the combined loading condition to 30 percent of the above values, strain gage data were reviewed. These data indicated that the maximum load in the skin near the stepped joint would exceed the ultimate allowable load of 2736 pound/inch. Therefore, the predicted failure load was reduced by 10 percent in order that the predicted ultimate loads on the box would match the internal loads as near the allowable as possible. The ultimate loads for the structural test of the box therefore were set at

$$M_x = 1,971,000 \text{ inch-pound}$$

$$T = 414,000 \text{ inch-pound}$$

$$V = 13,320 \text{ pound}$$

Tests were performed in the three phases shown below. Load directions are in accordance with Figure 10-5. All loads were applied in 10 percent increments to the values listed. Strain gage and deflection measurements were obtained at each increment. Prior to application of Phase III loads, the outer skins of the specimen were stabilized at the test temperature for a minimum of 5 minutes. Phase III strain gage, deflection, and temperature measurements were obtained at each 10 percent increment until failure.

Test Phase	Percent of Design Ultimate Load	Applied Loads			Test Temperature
		M(in.-lb)	T(in.-lb)	V(lb)	
I	50	985,500	-	-	Room
II	50	985,500	207,000	6,660	Room
III	100	1,971,000	414,000	13,320	+500 F

During Phase III test, a thread on a hydraulic jack failed, causing a halt in the test. Maximum load achieved was equivalent to 70 percent of the design ultimate. After repair of the jack, Phase III testing was restarted.

Failure of the beam occurred after a short hold at 80 percent of ultimate design load (120 percent of limit) when the secondary bond of the spar to compression cover parted. This failure, shown in Figure 10-8, initiated under the front spar load reaction fitting (see Figure 10-9) and proceeded in both directions along the bond line. Upon removal of the beam

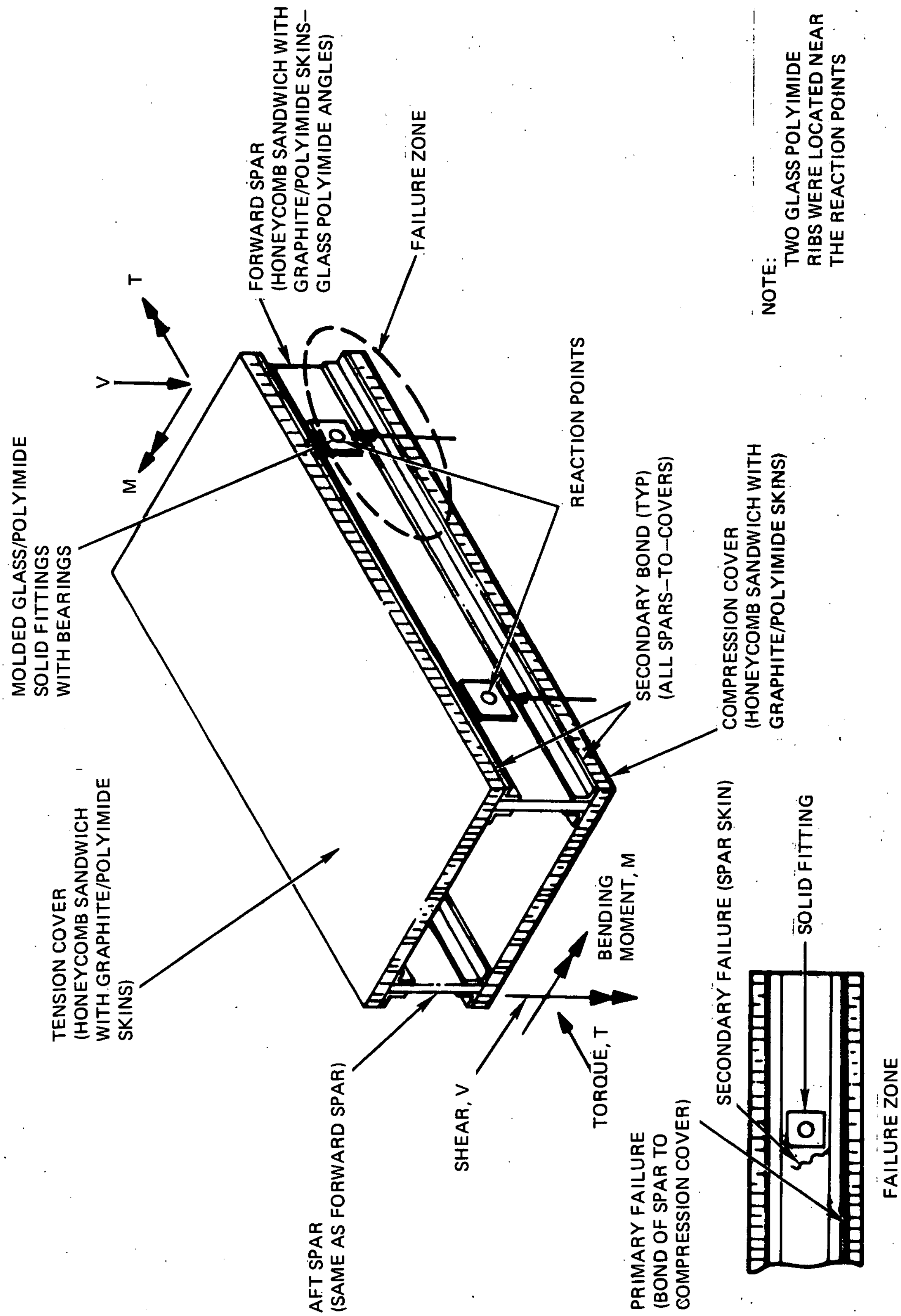


Figure 10-8. Graphite Polyimide Box Beam Test Results

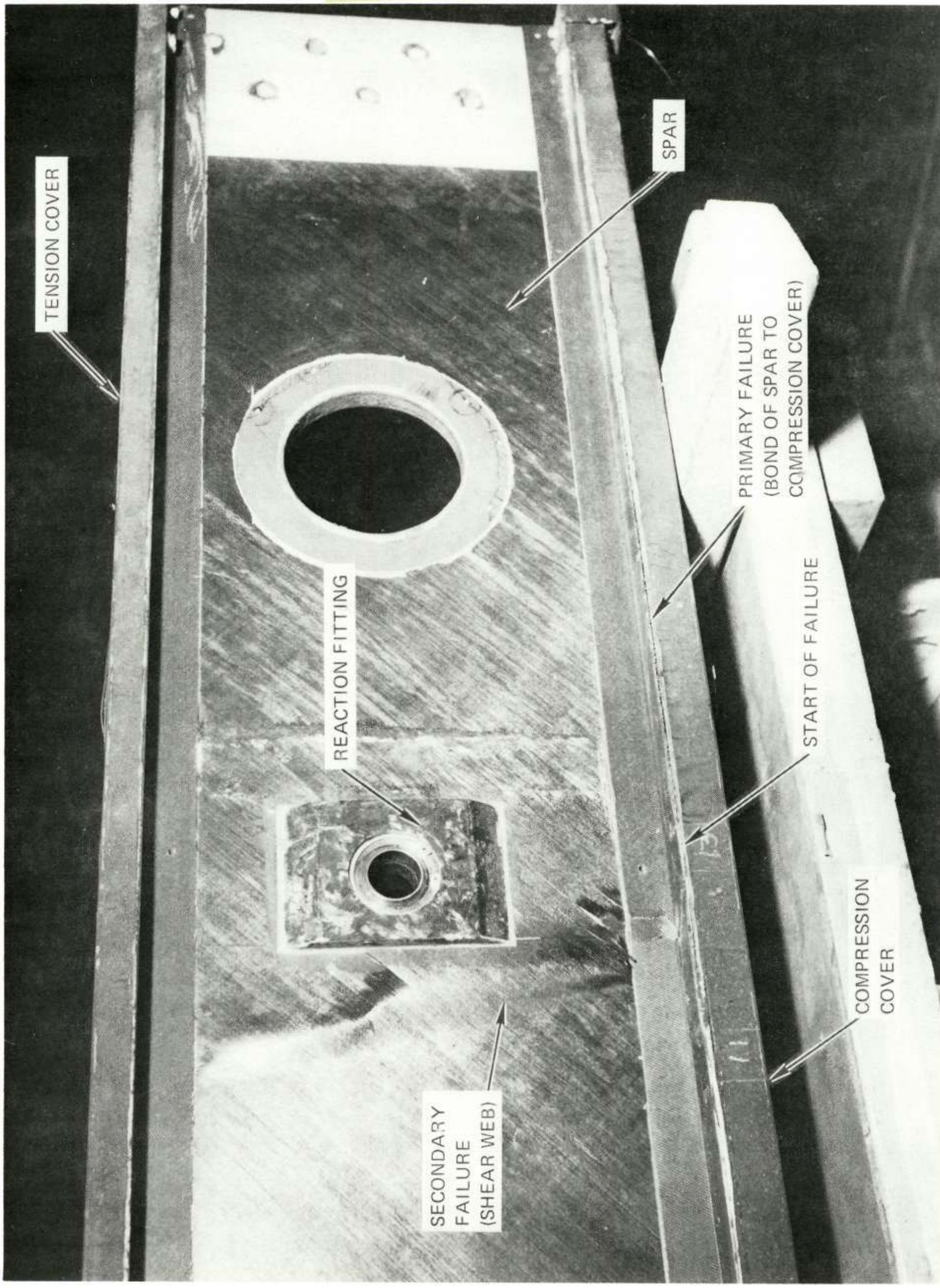


Figure 10-9. Graphite Polyimide Box Beam Failure

from the test fixture, a second failure zone was noted in the shear web of the spar immediately inboard of the fitting and proceeding in a direction approximately 7 degrees from the vertical.

10.2 TEST DATA

Strain gage and deflection data for room teperature tests (Phases I and II) are presented in Tables 10-1 and 10-2. A review of data plots indicated that deflections and strains behaved linearly. Maximum strain occurred in the compression cover skin adjacent to the access door (Gage A-1). This strain was -2130 μin./in., which is approximately equal to a stress of 26 ksi. Maximum strain at gages near the titanium bond was -1830 μin./in. (Gage R-1). This reduced to a compression stress of 19.6 ksi taking into account the biaxial strain.

Test data for Phase III second tests are shown in Tables 10-3 and 10-4. The last reading was at 70 percent of ultimate. All deflections and strains were linear with the exception of Gage R-7. Inspection of data from this gage indicates that the bond of the gage to the cover was questionable. Corner deflections of the beam were in close agreement with room temperature data. True strains at 30 percent of ultimate were within 10 percent of the room temperature strains. Maximum true strains, extrapolated to the 80 percent load level at failure, were 4380 μin./in. and 3580 μin./in. for Gages A-1 and R-1, respectively. These values included the thermal strain of -490 μin./in. and -400 μin./in. for the former and latter gages, respectively.

10.3 DATA ANALYSIS

Deflections of the box beam were measured during test phase at 10 percent increments of load. The maximum deflection occurred at the corner near the final failure zone. The 80 percent load level extrapolated data, based on Table 10-4, indicates a deflection of 0.522-inch at this point. This value is 22 percent greater than the deflection at the matching corner on the opposite end of the beam. Since one-half of this deflection ratio occurred in early room temperature tests and since checkout of deflectometers and load cells did not show any problem, the large deflection may be attributable, in part, to material variance (amount of fibers present) as noted in Section 9.1, Quality of Incoming Material. The greater ratio of corner deflections at elevated temperature could be the result of a resin dependency caused by collimation deficiencies. Failure of the beam was anticipated at the large deflection corner after initial tests. The exact mode of failure, however, was not predictable.

Table 10-1. Graphite Polyimide Box Beam Test
50% Design Ultimate Runs
Room Temperature
Strain in Microinches/Inch

Gage No. \ % Ultimate	Bending Only		Bending, Shear and Torsion	
	30%	50%	30%	50%
A-1	-920	-1570	-1280	-2130
A-2	+780	1280	900	1680
A-3 (Door)	-250	-510	-460	-650
A-4	780	1260	870	1480
A-5	-1010	-1700	-1260	-1890
A-6	770	1270	840	1350
R-1 (Fwd)	320	520	300	510
R-1 (Inb'd)	-880	-1420	-1100	-1830
R-1 (45°)	-310	-600	-220	-300
R-2 (Fwd)	280	530	300	490
R-2 (Inb'd)	-720	-1240	-710	-1480
R-2 (45°)	-400	-680	-200	-300
R-3 (Fwd)	-210	-410	-180	-320
R-3 (Inb'd)	-	-	-	-
R-3 (45°)	260	390	20	-30
R-4 (Fwd)	-230	-420	-310	-510
R-4 (Inb'd)	800	1300	940	1580
R-4 (45°)	-	-	-	-
R-5 (Fwd)	-	-	-	-
R-5 (Inb'd)	-730	-1280	-800	-1380
R-5 (45°)	-200	-400	-100	-130
R-6 (Fwd)	-230	-440	-310	-520
R-6 (Inb'd)	870	1440	920	1550
R-6 (45°)	310	510	180	290
R-7 (Fwd)	400	750	340	720
R-7 (Inb'd)	-730	-1220	-920	-1360
R-7 (45°)	-50	-30	80	230

Table 10-1. Graphite Polyimide Box Beam Test (Cont)

Gage No. \ % Ultimate	Bending Only		Bending, Shear and Torsion	
	30%	50%	30%	50%
R-8 (Fwd)	320	480	290	470
R-8 (Inb'd)	-710	-1220	-820	-1340
R-8 (45°)	30	30	50	90
R-9 (Fwd)	-150	-340	-200	-300
R-9 (Inb'd)	710	1120	700	1120
R-9 (45°)	-	-	-	-
R-10 (Fwd)	-210	-400	-420	-600
R-10 (Inb'd)	820	1280	890	1400
R-10 (45°)	200	280	30	60
R-11 (Up)	20	40	110	170
R-11 (Inb'd)	40	40	-10	-10
R-11 (45°)	40	40	10	80
R-12 (Up)	10	0	-80	0
R-12 (Inb'd)	-	-	-	-
R-12 (45°)	0	0	720	1210
R-13 (Up)	390	590	650	1080
R-13 (Inb'd)	-330	-610	-220	-290
R-13 (45°)	0	-40	-150	-210

Note: Refer to Figure 10-1 for Location of Gages

Table 10-2. Graphite Polyimide Box Beam Test
50% Design Ultimate Runs
Room Temperature

Gage No. \ % Ultimate	Deflection in Inches			
	Bending Only		Bending, Shear and Torsion	
	30%	50%	30%	50%
1	-0.016	-0.016	+0.006	+0.008
2	-0.016	-0.016	+0.006	+0.010
3	+0.062	+0.122	+0.150	+0.258
4	+0.062	+0.124	+0.070	+0.120
5	-0.018	-0.018	+0.004	+0.010
6	-0.012	-0.012	-0.002	-0.004
7	-0.022	-0.024	+0.004	+0.004
8	-0.022	-0.026	-0.032	-0.040
9	-0.020	-0.026	-0.008	-0.018
10	-0.030	-0.044	-0.028	-0.044
11	-0.016	-0.016	+0.004	+0.020
12	-0.020	-0.024	-0.020	-0.022
13	-0.018	-0.018	+0.002	+0.016
14	-0.018	-0.018	-0.008	-0.008
15	+0.074	+0.134	+0.180	+0.302
16	+0.074	+0.144	+0.094	+0.170

Note: Refer to Figure 10-2 for Location of Gages

Table 10-3. Graphite Polyimide Box Beam Test
Final Run
Load and Temperature
Strain in Microinches/Inch

Gage No. \ % Ultimate	Strain Readings: 0 Load 5 Min @500 F	Strain Readings: 30% Load 500 F	Strain Readings: 70% Load 500 F	Temperature (F): 1*	Temperature (F): 2*	True Strain: 0 Load 5 Min @500 F	True Strain: 30% Load 500 F	True Strain: 70% Load 500 F
A-1*1	-480	-1840	-3790	484	478	-490	-1870	-3840
A-2	-600	400	1780	504	510	-610	410	1810
A-3	-600	-790	-1380	491	495	-610	-800	-1400
A-4	-650	190	1320	491	486	-660	190	1340
A-5	-600	-1750	-3310	484	478	-610	-1780	-3360
A-6	-520	290	1370	504	510	-530	300	1390
R-1 (Fwd)	150	460	930	471	468	150	470	950
R-1 (Inb'd)	-390	-1540	-3090	471	468	-400	-1560	-3140
R-1 (45°)	-240	-390	-580	471	468	-240	-400	-590
R-2 (Fwd)	290	600	1010	250	285	330	640	1060
R-2 (Inb'd)	20	-960	-2190	250	285	60	-930	-2160
R-2 (45°)	150	-40	-280	250	285	190	0	-240
R-3 (Fwd)	600	390	100	304	334	640	430	140
R-3 (Inb'd)	-	-	-	304	334	-	-	-
R-3 (45°)	340	310	280	304	334	380	350	320
R-4 (Fwd)	-240	-520	-900	456	447	-220	-510	-890
R-4 (Inb'd)	-640	350	1640	456	447	-630	370	1680
R-4 (45°)	-	-	-	456	447	-	-	-
R-5 (Fwd)	-	-	- $\boxed{1}$	478	476	-	-	-
R-5 (Inb'd)	-400	-1210	-2010 (60%)	478	476	-410	-1230	-2040
R-5 (45°)	-40	-130	-180 (60%)	478	476	-40	-130	-180
R-6 (Fwd)	20	-220	-600	476	478	20	-220	-610
R-6 (Inb'd)	-850	140	1420	476	478	-860	140	1440
R-6 (45°)	-270	-80	130	476	478	-270	-80	-130

Note:

1 Gage R-5 out at 70% ultimate

* Reading #1 taken 5 min after 500°F; #2 at 500°F and 70% load

*1 A-1 was read just before failure (-4810 true μe)

*2 Gage R-7 values are questionable (possible bad gage)
Refer to Figure 10-1 for strain gage locations and Figure 10-3 for thermocouple locations.

Table 10-3. Graphite Polyimide Box Beam Test (Cont)

Gage No. \ % Ultimate	Strain Readings			Temperature (F)		True Strain		
	0 Load 5 Min @500 F	30% Load 500 F	70% Load 500 F	1*	2*	0 Load 5 Min @500 F	30% Load 500 F	70% Load 500 F
R-7 (Fwd)*2	270	1100	4010	471	468	270	1120	4080
R-7 (Inb'd)*2	-370	-1060	-630	471	468	-370	-1070	-640
R-7 (45°)*2	130	540	2450	471	468	130	550	2490
R-8 (Fwd)	310	600	1000	250	285	350	640	1050
R-8 (Inb'd)	0	-870	-1970	250	285	40	-830	-1940
R-8 (45°)	300	380	470	250	285	340	420	510
R-9 (Fwd)	390	200	-90	304	334	430	240	-50
R-9 (Inb'd)	-10	680	1510	304	334	30	720	1550
R-9 (45°)	-	-	-	304	334	-	-	-
R-10 (Fwd)	20	-250	-550	456	447	40	-230	-530
R-10 (Inb'd)	-600	240	1300	456	447	-590	260	1330
R-10 (45°)	30	100	150	456	447	50	120	170
R-11 (Up)	10	70	300	121	206	30	100	340
R-11 (Inb'd)	40	80	-30	121	206	60	110	10
R-11 (45°)	20	-10	-20	121	206	40	20	20
R-12 (Up)	90	70	180	118	158	110	90	210
R-12 (Inb'd)	-	-	-	118	158	-	-	-
R-12 (45°)	90	890	1900	118	158	110	910	1940
R-13 (Up)	560	1180	2060	118	158	580	1200	2090
R-13 (Inb'd)	160	90	50	118	158	180	110	80
R-13 (45°)	10	-80	-180	118	158	30	-60	-150

Note:

1 Gage R-5 out at 70% ultimate

* Reading #1 taken 5 min after 500°F; #2 at 500°F and 70% load

*1 A-1 was read just before failure (-4810 true μe)

*2 Gage R-7 values are questionable (possible bad gage)
Refer to Figure 10-1 for strain gage locations and
Figure 10-3 for thermocouple locations.

Table 10-4. Graphite Polyimide Box Beam Test
Final Run
Load and Temperature
Deflection in Inches

Gage No. \ % Ultimate	0 Load 5 Min at 500 F	30% Load 500 F	70% Load 500 F
1*	+0.002	+0.004	+0.008
2*	0	+0.002	+0.006
3	0	+0.142	+0.374
4	-0.002	+0.066	+0.162
5	0	+0.008	+0.032
6	-0.002	-0.004	-0.010
7	0	+0.006	+0.014
8	+0.002	-0.010	-0.026
9	0	-0.006	-0.018
10	0	-0.018	-0.052
11	0	0	+0.006
12	+0.002	-0.002	-0.006
13	+0.002	+0.004	+0.030
14	0	-0.002	-0.004
15	0	+0.196	+0.456
16	0	+0.090	+0.212

Note:

* Gages 1 and 2 were located on the reaction jig at 1.5 inches from front spar centerline.

1 + Deflection is up

2 Refer to Figure 10-2 for gage locations

The mechanical strains at the 500 F failure load were extrapolated from 70 percent of load data (Table 10-3). The maximum strain occurred at Gage A-1. Based on a calculated unidirectional mechanical strain of -3840 μin. /in., the compression stress was approximately 40 ksi. This stress, which is equivalent to 2520 lb/in., is approximately 90 percent of the minimum compressive strength measured in laboratory tests.

The mechanical strain in the section of the cover at a distance from the access door is calculated from Table 10-3 to be -2740 μin. /in. and +2260 μin. /in. for the compression and tension covers, respectively. These strains are equivalent to stress levels of -32. 5 ksi and 26. 9 ksi. The compression level is approximately 60 percent of the failure stress measured in the stepped lap structural element. The tension cover was at a level equal to 50 percent of the stress in the element. Strain gages for these data were located at the opposite end of the box beam from the failure.

C

11.0 NEW TECHNOLOGY

11.1 TECHNOLOGY UTILIZATION ITEMS

The following Technology Utilization items are credited to the G/PI Box Beam Program and were reported during its course:

T. U. - SD No.	Title
94268	Autoclave Modification for Curing Polyimide Adhesive Bonding of RP Structure
94420	Development of Co-Cure Bonding Processes for Integrally Joining Graphite Fiber Reinforced Polyimide Composite to Titanium Alloy
94001	Technical Paper, Space Shuttle Oriented Polyimide Matrix Composite Studies
94016	Increase Transparency of Close Mesh Wire Screens
94017	Obtaining Smooth Surface with High Temperature Curing Plastic Laminates
94018	Protection of Glass with Teflon Film
94020	Postforming of PI Glass Laminate
94021	Fabrication of Polyimide Laminate Tubing
94106	Felted Paper Bleeder Material for Molding Composite Laminates
93644	A Method for Molding Non-Metallic Composites Requiring High Heat Rise Cures

T. U. - SD No.	Title
93535	Determination of Graphite/Polyimide Constituent Fractions by Parametric Graphical Methods
93536	Elevated Temperature Interlaminar Shear Testing
93560	Containment of High Temperature Insulation

11.2 MODMOR II/POLYQUINOXALINE STUDIES

Processing studies were carried out on the Modmor II/Polyquinoxaline prepreg furnished by NASA/MSFC. A total of nine panels was laminated in these studies. The following is the cure cycle established by these studies.

Staging

1. Stack prepreg layers on an open face platen that allows venting of the preform from the bottom.
2. Vent the top through dead weighted 1/8 inch cell aluminum honeycomb core.
3. Place assembly stack in 395 to 400 F preheated air circulating oven.
4. Heat to 400 F in 4 minutes.
5. Stage 5 minutes at 400 F and remove from oven.

Molding

1. Preheat press to 750 ± 10 F.
2. Load stacked preform into R. T. mold.
3. Load mold in press and apply contact pressure.
4. Apply 250 psi pressure when part reaches 320 F.
5. When part reaches 630 F bump mold and reapply 250 psi pressure.

6. When part reaches 750 ± 10 F cure for 5 hours at 750 F and 250 psi.

7. Cool to 400 F prior to releasing pressure.

Post Cure

1. Place laminate in a retort connected to a nitrogen source and purge at approximately 0.3 CFM throughout post cure.

2. Raise to 750 ± 10 F and post-cure for 14 hours.

3. Maintain nitrogen purge during cooling down to 300 F.

Essentially the same results can be obtained by continuing the 750 ± 10 F cure cycle for a total of 19 hours and eliminating the post cure cycle.

This cure cycle varies from that recommended by the vendor, Whittaker R&D, in the following respects.

1. The material was debulked by staging in an oven prior to the cure cycle.

2. Pressure was applied during cure at 320 F rather than at the end of the period at which the material had stopped smoking (usually 2-3 minutes).

3. The pressure used during cure was 250 psi rather than 500 psi.

4. The laminates were post cured.

Physical properties and test results from panels laminated using the developed cure cycle are presented in Table 11-1.

11.3 OTHER RELATED TECHNOLOGY

Specific technology efforts and developments deemed to be of particular interest due to their intimate relationship to the G/PI box beam program are described below.

Miscellaneous Polyimide Resins

Graphite/Sablon laminates were received from Narmco Materials Division and tested at RT. The short beam shear strength was only 6.5 ksi.

Table 11-1. Modmor II/Polyquinoxaline

Physical Properties

Panel No.	Density Lb/Cu In.	Resin Content Vol %	Fiber Content Vol %	Void Content Vol %	Thickness Mils
PQ8	0.057	31.9	61.2	1.4	7.9
PQ9	0.056	33.0	58.9	2.9	8.2

Mechanical Properties*

Panel No.	Flexural Strength Long. (Ksi)		Flexural Modulus Long. (MSI)		Flexural Strength Trans. (Ksi)		Tensile Strength Long. (Ksi)		Tensile Modulus Long. (MSI)		Tensile Strength ± 45 (Ksi)		Short Beam Shear Long. (Ksi)	
	RT	600	RT	600	RT	600	RT	600	RT	600	RT	600	RT	600
PQ8	252	149	18.9	17.3	9.0	5.0	146	146	18.1				12.4	5.5
PQ9											16.8	14.0		

*Average of 3 tests

Evident low density and high void content appear connected with excessive resin runout during cure.

A Modmor II/Kerimid 601 panel molded by the Process 14 for Gemon L showed good (13 ksi) RT short beam shear strength; however, 500 F tests resulted in only 2.8 ksi shear strength and severe plastic deformation. Kerimid 601 is the Rhone-Poulenc resin on which Gemon L is based.

Elevated Temperature Shear Test

A review of preliminary 600 F test data of graphite/polyimide short-beam shear specimens indicated that in many cases room temperature (RT) methods for failure load definition cannot be employed (i. e., using the load drop-off point of the test machine indicator dial as a measure of shear failure). Load-versus-crosshead travel charts of these tests, however, show that reasonable ultimate shear strength comparisons may be made by using an off-set deflection technique. This technique permits the definition of the initial failure when it does not produce a definite drop-off in load. Although the test machine operator may be able to pick up the change in load rate when a constant crosshead speed is used, considerable scatter may be expected in the test results. The described methods reduce this scatter and produce a permanent record for future reference.

Data from the four different panels used in a 600 F preliminary study indicate that differences in processes and matrix materials can be observed by this approach:

Panel No.	Material	2% Offset Yield Shear Strength, Ksi Range	Ave
PD-25-1-13	Dupont 4707 Courtauld HTS	3.5-5.3	4.6
PD-24-1-13*	Dupont 4707 Courtauld HTS	3.7-4.1	3.9
PS-1-2-16	Skybond 703 Modmor II	3.7	3.7
PG-3-1-14	Gemon-L Courtauld HTS	2.2-3.6	2.7

The method of establishing the load at which yield shear failure occurs is shown graphically in Figure 11-1. Briefly, (1) the apparent initial slope of the load-deflection plot is established, (2) a line parallel to the load deflection slope is drawn at a distance equivalent to an incremental crosshead travel, and (3) the load at the intersection of the load-crosshead travel curve and the offset line is assumed to be the yield load of the specimen .

The off-set yield shear strength of the specimen is calculated using this load and standard equations. The recommended off-set distance and equations for shear strength are shown on the figure.

It should be pointed out that any interlaminar shear technique is merely a means of judging relative merits of similar materials, since the calculated strength is configuration sensitive. Therefore, the described method of establishing the short beam shear strength produces relative values and thus should be used only for screening and comparison purposes. It is also pointed out that in cases and at temperatures where this method has to be applied, the overall mechanical properties are generally too low to be of real interest.

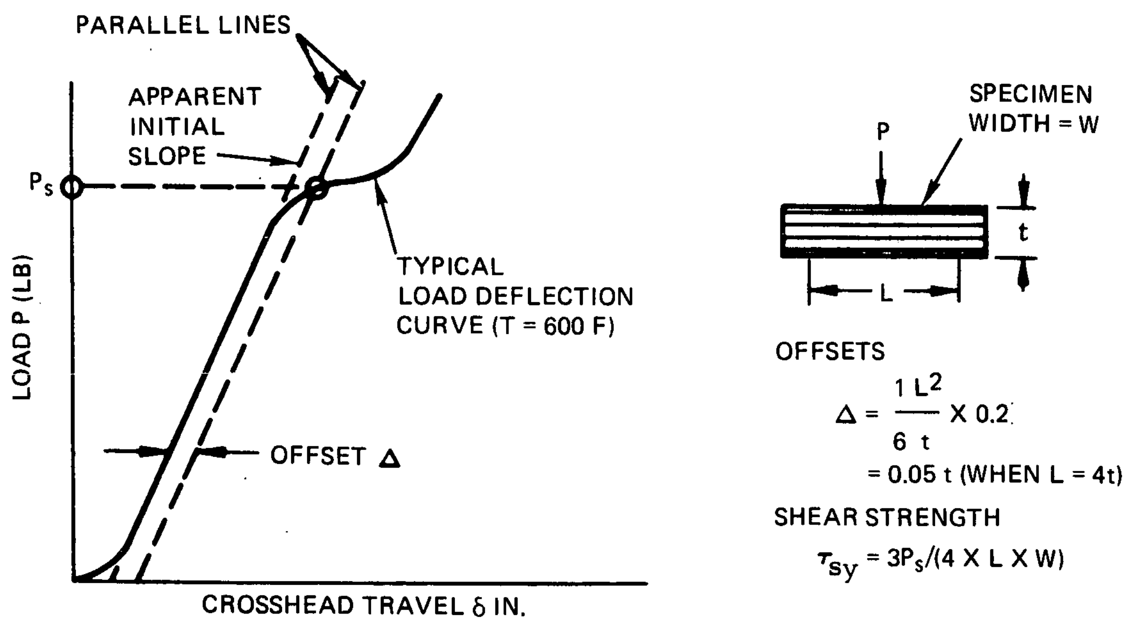


Figure 11-1. Offset Short Beam Yield Shear Strength Method

Graphical Determination of Fiber and Void Content

Parametric graphical methods were developed which require only simple measurements for reasonably accurate estimation of constituent fractions. An example is given below for Modmor II/Gemon L.

The fiber content of the cured laminate in volume percent can be read off from the information provided in Figure 11-2. The average prepreg fiber weight per ply per unit area is known from determinations on the prepreg and is assumed to remain constant in the laminate. The average ply thickness can be simply determined by micrometer measurement, dividing the laminate thickness by the plies used. The curves relating the fiber volume percentage to the prepreg unit weight for the various ply thicknesses have been derived for the Modmor II density, ρ_f = 0.0635 lb/cu. in. from the following simple relation:

$$\overline{V}_f = \frac{W_f}{454 \times 144 \times \rho_f \times t_1} \times 100$$

where:

$\overline{V}_f$ = Volume of fiber in laminate

W_f = Weight of fiber in prepreg, gm/sq ft

ρ_f = Fiber density, lb/cu in.

t_1 = Ply thickness of cured laminate

Figure 11-3 permits estimation of the void content in volume percent. The fiber content, known from Figure 11-2, is plotted against laminate density (a simple determination) for a series of constant void contents from the relation:

$$\rho_1 = \frac{\overline{V}_f \ \rho_f + \overline{V}_r \ \rho_r}{100}$$

The constituent fractions are related by

$$\overline{V}_r = 100 - \overline{V}_f - \overline{V}_V$$

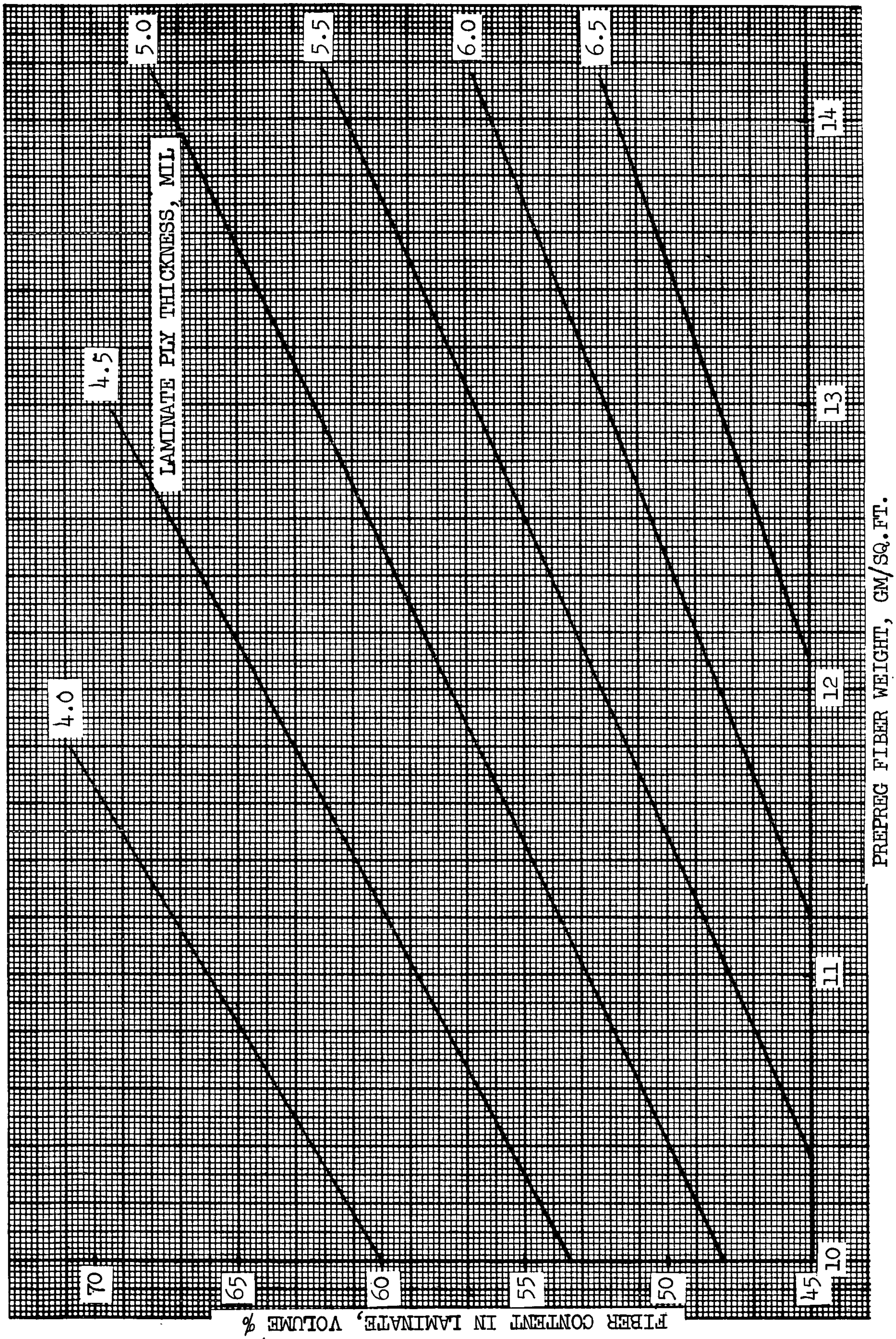


Figure 11-2. Fiber Content, Cured Laminate Versus Prepreg Fiber Weight

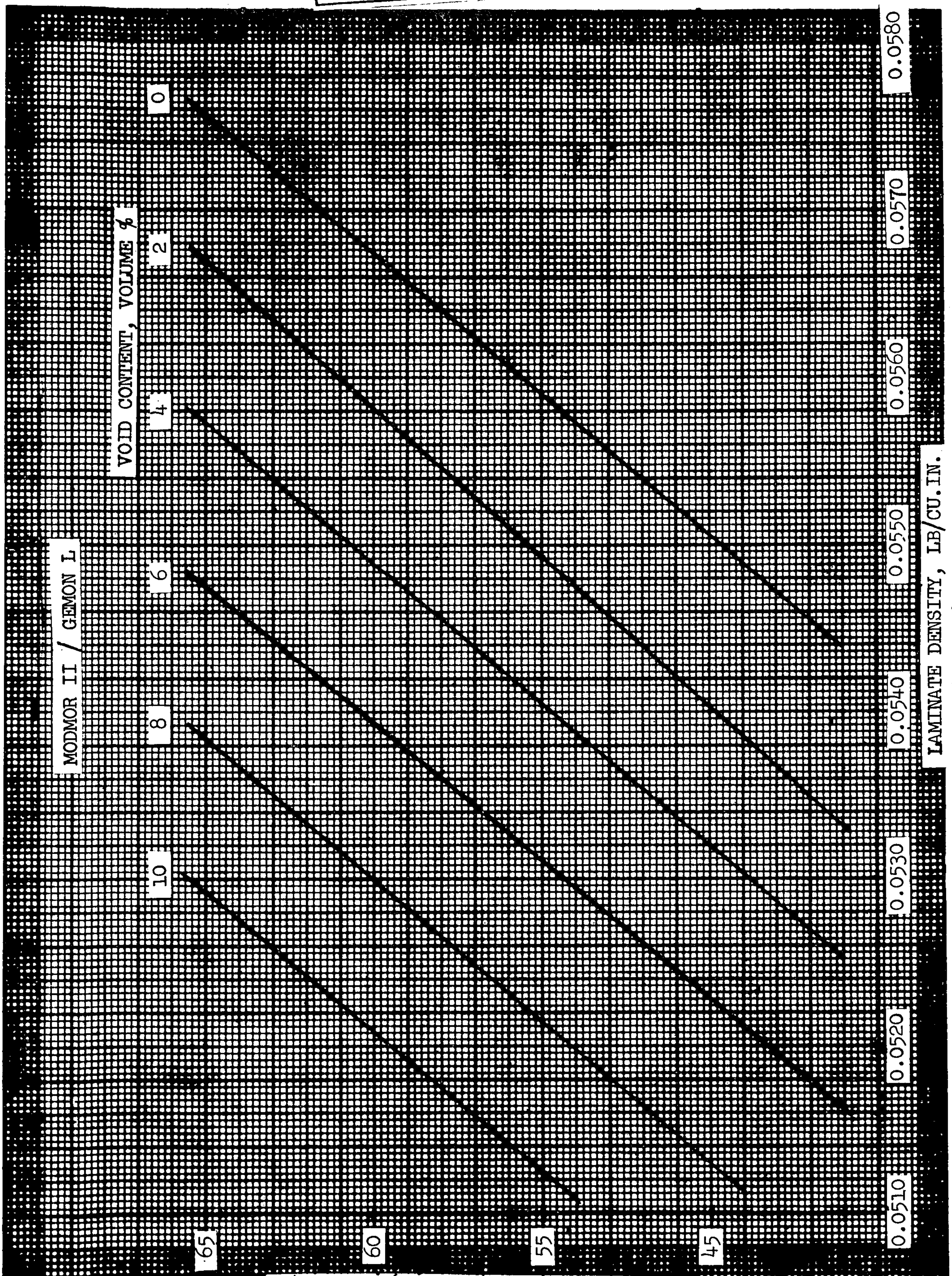


Figure 11-3. Laminate Density Versus Fiber Volume Percent for Constant Void Contents

Symbols used are:

V = Constituent content, volume percentage

ρ = Density, lb/cu in.

and subscripts

l = laminate

f = fiber

r = resin

V = voids

ρ_r = 0.046 lb/cu in. was used in Figure 11-3, representative of Gemon L.

Information derived in the fashion described is sufficiently accurate for component program purposes, provided uniformity of the prepreg material is within specified boundaries.

Narmco 2056 Adhesive

A sample of this high temperature adhesive was received from Whittaker Research and Development Corporation for evaluation. It was stated to be produced from a new polymer by a hot melt method, to contain aluminum filler, and to produce zero volatiles in cure. Lap shear tests on titanium adhered specimens bonded with this material were performed at RT, 500 F, and 550 F. The results of these tests, shown in Table 11-2, are very respectable. The specimen configuration, surface preparation, and cure cycle are also shown in this table. The higher bond strength at 500 F coupled with the 100 percent solids characteristics make this adhesive a strong candidate for low pressure bonding of structures designed to operate at this temperature. For a time, it was considered as back-up for the final assembly of the box beam subassemblies.

Table 11-2. Tensile Shear Strength of Narmco 2056 Adhesive, Ti 6Al-4V Adherends, Doublt Lap, 0.6-IN. O.L. 1)

	RT	500F	550F
Tensile Shear Strength, ksi	3642	2700	1488
	3833	2536	1448
	4062	2529	
	3847	2588	1468

1) Adherends

Cleaned per MA0110-024 plus liquid hone, no primer employed. Specimens bonded in press at 40 psi, 350 F for 1 hour. Heat rise rate approximately 4.5 F per minute RT to 350 F.

All failures cohesive

12.0 CONCLUSIONS AND RECOMMENDATIONS

This manufacturing technology program developed the designs, processes, quality control procedures, specifications, and tooling concepts necessary to fabricate large structural components from Modmor II/Gemon L or other advanced composite materials. As a result of this program, the following conclusions are drawn:

1. Gemon L resin provides a matrix material for advanced fiber reinforced composite with a combination of ease of processing and 500 F service temperature. Although this resin is currently out of production, an analogue is available, produced by Rhone-Poulenc, France, under the tradename of "Kerimid 601."[1]

2. Large sheet-type broadgood materials pose inspection, storage, and handling problems. The control of the areal fiber weight of broad goods is of particular importance. The selection and rejection of portions of sheet goods, which can be employed for development hardware, would not be an acceptable procedure for production. Based on this observation, tape goods are preferred.

3. Current polyimide high temperature adhesives must be cured under vacuum in order to eliminate volatiles and thus obtain a reliable bond.

4. Co-cure of bonding adhesives and laminate resins has been proven for the selected system.

5. The major problems in composite structures are concerned with the detail design of concentrated load points, mechanical joints, and secondary bonds.

As a result of this program, the following items are recommended for study in order to achieve reliable high temperature advanced composite aerospace structures:

1. Develop addition type polyimides which can be cured with standard manufacturing equipment. Production autoclaves currently are limited to approximately 400 F although some laboratory autoclaves can go to 550 F and 800 F.

1) U.S. Distributor: Rhodia Corporation, New York City, N.Y.

2. Establish processes that can consistently produce reliable large components of condensation-type polyimides.

3. Develop co-cured adhesive/laminate processes for the above resins.

4. Develop high temperature adhesives having 100 percent solids content in order to minimize fabrication costs. Such adhesive studies would simplify secondary bonding processes by the eliminating of the necessity to remove volatiles.

5. Define design techniques for combined mechanical and bonded joints between components for utilization in areas of high shear concentrations.

6. Fabricate and test large structural components to verify that "real world" processes have been developed. The program scope for these articles should include detail structural analyses of all critical areas, process definition, and structural element fabrication and test.

13.0 REFERENCES

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2. Design, Fabrication, and Testing of Glass-Fiber Reinforced-Plastic Box Beam Structures, 2 Spar Configuration. North American Rockwell Corp., Columbus Div., Rpt. NADC-ST-7004 (March 1970).

3. Clark, G. A. and K. I. Clayton, North American Rockwell Corp., Columbus Div. - "Fabrication Techniques for Advanced Composite Attachments and Joints," Techn. Rpt. AFML-TR-69-151.

4. Clark, G. A. and K. I. Clayton, Titanium Reinforced Boron Polyimide Composite. North American Rockwell Corp., Columbus Div. - Final Report NR71H-511 (27 June 1969).

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6. Optimization and Evaluation of High Temperature Adhesives. North American Aviation, Inc., Los Angeles Division, Reports NA-65-1053, NA-66-459, and NA-66-467.

7. Saturn S-II Advanced Technology Studies, Study 1, Application of Composites on S-II Cross Beam, North American Rockwell Corporation, Space Division, SD 72-261 (February 15, 1972) Contract NAS7-200, Supplemental Agreement 2049.

APPENDIX A. PROCESSING CYCLES, PI PROCESSING, AND RESIN SELECTION[1] STUDIES

PROCESS NO. 1 (Pyralin 4707, 40 percent resin solids by weight)

1. Stage each face of required prepreg plies at 200 F for 75 minutes.

2. Prepare layup and bag as shown in Figure A-1. Apply full vacuum. Raise temperature to 240 F in 60 minutes.

3. Stage under full vacuum at 240 F for 20 minutes.

4. Maintain full vacuum and apply 90 psig autoclave pressure. Raise temperatue to 350 F in 30 minutes.

5. Hold at 350 F under vacuum and 90 psig pressure for 90 minutes.

6. Cool to 150 F under pressure and vacuum before withdrawing from autoclave.

7. Post-cure between restraining steel sheets in accordance with the following schedule:

   350 F for 40 minutes
   500 F for 2 1/2 ± 1/4 hour
   600 F for 4 ± 1/2 hour

8. Cool to 150 F before removing restraining steel sheets.

PROCESS NO. 2 (Pyralin 4707, 40 percent resin solids by weight)

1. Prestage in accordance with Step 1, Process No. 1.

2. Prepare, layup, and bag as shown in Figure A-2. Apply 90 psig autoclave pressure and maintain this basic pressure for remainder

[1] Unless specifically stated otherwise, tolerances on cure parameters are as follows: time: + 2 minutes; holding temperature: ±5 F; temperature rise rate; ±10 percent; vacuum: 24 inches of Hg minimum; autoclave pressure: ±5 psig.

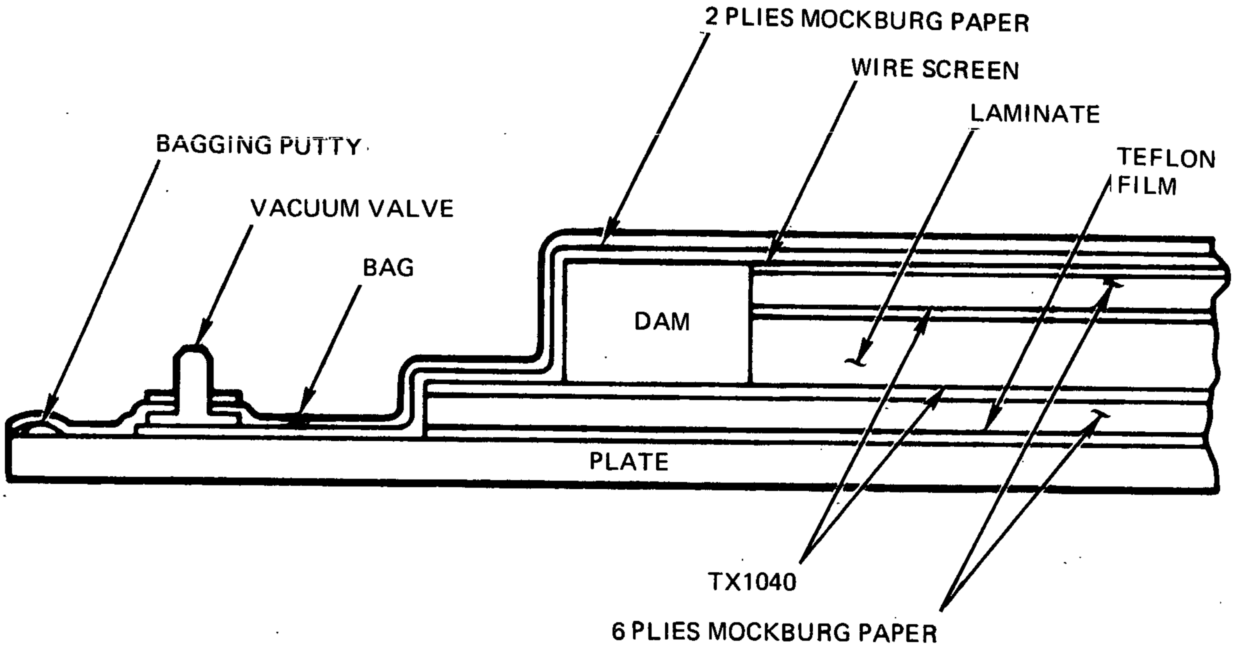

Figure A-1. Layup Diagram, Pyralin 4707 Standard Process

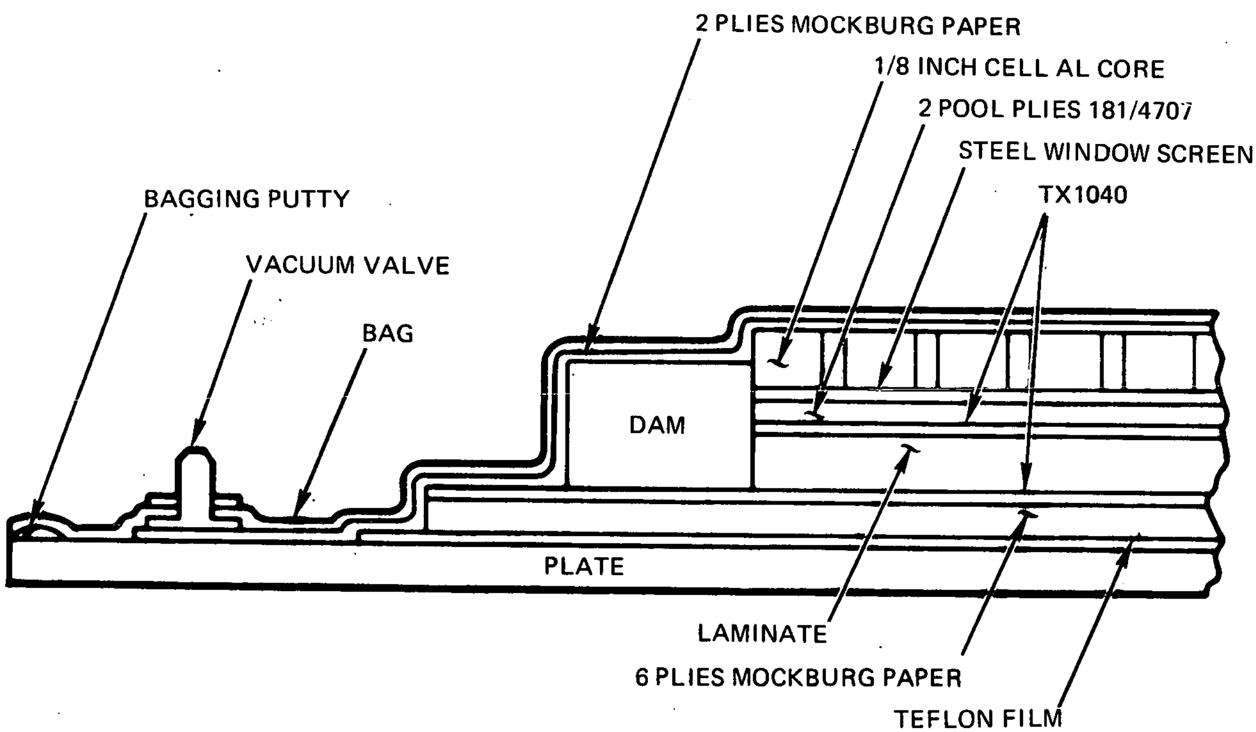

Figure A-2. Layup Diagram, Pyralin 4707 Pressure Cycle Process of cure. Start alternating pressure cycle in accordance with the following schedule:

30 seconds off - 10 psig
2 minutes on - 90 psig plus vacuum

Note: The lower pressure during the "30 seconds off" period is achieved by bleeding 80 psi pressure into the bag.

3. Raise temperature to 240 F for 20 minutes.

4. Terminate alternating pressure cycle. Maintain 90 psig plus full vacuum for remainder of cure. Raise temperature to 350 F in 30 minutes.

5. Proceed in accordance with Steps 5 through 8, Process No. 1.

PROCESS NO. 3 (Pyralin 4707, 55 percent resin solids by weight)

1. Soak individual prepreg plies in circulating water for 6 minutes ± 15 seconds. Blot surface. Trim.

2. Prepare layup and bag as shown in Figure A-3. Proceed with rest of processing cycle in accordance with Steps 2 through 8, Process No. 1.

PROCESS NO. 4 (Gemon L)

1. Prepare layup and bag as shown in Figure A-4. Load into autoclave and apply 5 psig positive pressure, the layup being vented to the atmosphere (i. e., no applied vacuum). Raise temperature to 250 F at a rate of 3 F per minute.

2. Hold at 250 F for 50 minutes, maintaining 5 psig, with layup vented to atmosphere.

3. Raise applied pressure to 90 psig, maintaining atmosphere venting. Raise temperature to 350 F in 30 minutes.

4. Hold at 350 F for 90 minutes.

5. Cool to 150 F under 90 psig, atmosphere venting, before withdrawing from autoclave.

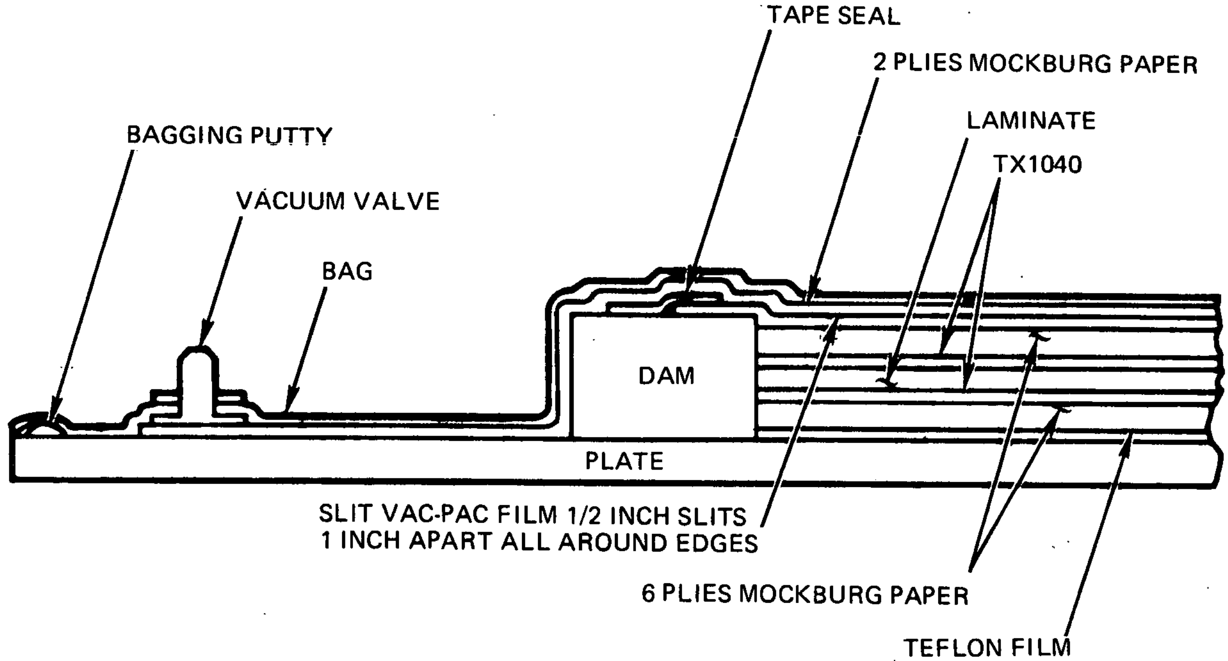

Figure A-3. Layup Diagram, Pyralin 4707 Water Soak Process

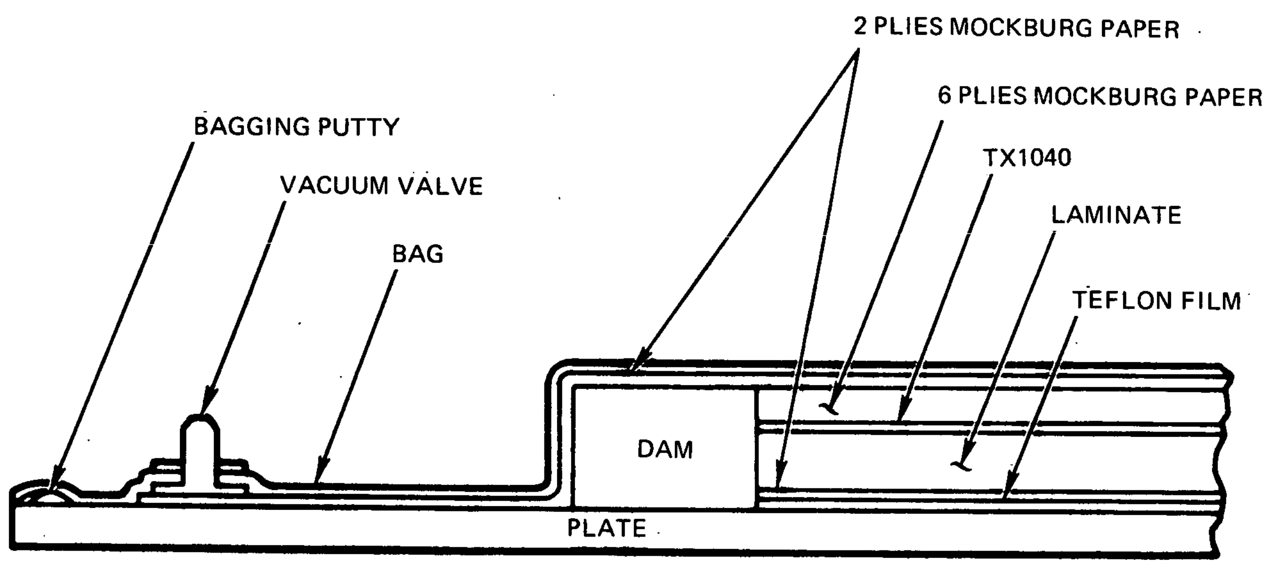

Figure A-4. Layup Diagram, Gemon L One Stage Process

6. Post-cure between restraining steel sheets according to the following schedule:

   6 hours at 300 F
   1 hour at 350 F
   1 hour at 400 F
   1 hour at 450 F
   1 hour at 482 F

   Note: The heating rate between holding levels is not critical; 5 to 10 minutes is suggested to reach the next higher level.

7. Continue post-cure by raising temperature from 482 F to 600 F at a rate of 2 F per hour. Hold at 600 F for 2 hours.

8. Cool to 150 F before removing restraining steel sheets.

PROCESS NO. 5 (Gemon L)

Proceed identically to Process No. 4, except substituting 565 F for 600 F in Step 7, Continuation of Post-Cure.

PROCESS NO. 6 (Gemon L)

1. Prepare layup and bag as shown in Figure A-4. Load into autoclave and apply 15 psig positive pressure, the layup being vented to the atmosphere (i. e., no applied vacuum). Raise temperature to 284 F at a rate of 6 to 8 F per minute.

2. Apply full vacuum. Continue raising of temperature at 6 to 8 F per minute to 305 F.

3. Increase applied pressure to 90 psig, also maintaining full vacuum. Continue raising temperature at a rate of 6 to 8 F per minute until 350 F ± 5 F is reached.

4. Hold at 350 F ± 5 F for 90 minutes.

5. Cool to 150 F under 90 psig and full vacuum before withdrawing from autoclave.

6. Post-cure between restraining steel sheets according to the following schedule:

   300 F for 12 hours
   350 F for 12 hours
   400 F for 6 hours
   450 F for 6 hours
   500 F for 6 hours

   Raise temperature from 500 F to 600 F at a rate of 1F every 5 minutes; hold at 600 F for 15 minutes.

   Note: The heating rate between holding levels is not critical; 5 to 10 minutes is suggested to reach the next higher level.

7. Cool to 150 F before removing restraining steel sheets.

PROCESS NO. 7 (Gemon L)

Proceed exactly as in Process No. 6, except for substituting 565 F for 600 F in Step 6, Post-Cure.

PROCESS NO. 8 (Gemon L)

Proceed as in Process No. 6 with the following exceptions:

A. Follow Steps 1 through 5 for successive partial layups of 0.025 to 0.030-inch thickness (three stages) in lieu of a single-stage layup.

B. Omit the bottom Mockburg paper bleeder plies.

C. Utilize a peel ply of F-56 (Coast Aerospace Materials, Hexcel Corporation) or equivalent, in lieu of TX 1040 and Mockburg paper for a combination of release, bleeding, and bond preparation purposes on top of the individual layups. The respective intermediate peel ply is to be removed from the preceding cured stack before commencing layup of the faying subsequent one.

PROCESS NO. 9 (Gemon L)[1]

Proceed as in Process No. 7 except for also utilizing the successive partial layup procedure of Process No. 8.

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[1] Panel not fabricated due to lack of sufficient material.

PROCESS NO. 10 (P13N)

1. Stage each face of required prepreg plies at 200 F for 20 minutes.

2. Stack layup between confining Coroprene dams (6 plies of Mockburg paper bleeder with TX 1040 separator) and stage under 10 inches of Hg vacuum at 350 F for 30 minutes. Raise vacuum to a minimum of 26 inches of Hg and hold for an additional 60 minutes.

3. Cool to resin temperature.

4. Apply aluminum foil parting agent to bottom of compression mold cavity.

5. Preheat compression mold to 550 F to 600 F.

6. Transfer prestaged layup into the mold cavity.

7. Using aluminum foil also as top parting agent, dwell layup one minute in tool with no pressure other than the dead weight of the plunger.

8. Cure layup at 575 F ± 25 F for 90 minutes under 500 psi pressure.

9. Remove layup hot.

PROCESS NO. 11 (Gemon L)

1. After material conditioning, prepare layup and bag and cure in accordance with Process No. 4 with the following exception:

   Interpose a 0.06-in.-thick aluminum caul plate between the outer 6-ply paper bleeder and 2 plies of paper breather.

2. Post-cure between restraining steel sheets according to the following schedule:

   300 F for 14 hours
   350 F for 12 hours
   400 F for 36 hours
   450 F for 36 hours
   500 F for 12 hours

Note: The heating rate between holding levels is not critical; 5 to 10 minutes is suggested to reach the next higher level.

3. Continue post-cure by raising temperature from 500 F to 565 F at a rate of 1 F every 5 minutes. Hold at 565 F for 15 minutes.

4. Cool to 150 F before removing restraining steel sheets.

PROCESS NO. 12 (Gemon L)

Proceed as in Process No. 11, except for omitting the bottom paper bleeder plies from the layup.

PROCESS NO. 13 (Gemon L)

1. After material conditioning, prepare layup and bag and cure in accordance with Process No. 6 (Progress Report No. 2) with the following exception:

   Interpose a 0.06-in.-thick aluminum caul plate between the 6-ply paper and the outer 2 plies of paper bleeder.

2. Post-cure including post cure continuation between restraining steel sheets in accordance with Process No. 11.

PROCESS NO. 14 (Gemon L)

Proceed as in Process No. 13, except for omitting bottom paper bleeder plies from layup.

PROCESS NO. 15 (Gemon L)

1. Prepare layup and bag as shown in Figure A-5. Load into autoclave and apply 5 psig positive pressure, the layup being vented to the atmosphere (i.e., no applied vacuum). Raise temperature to 284 F at a rate of 6 F to 8 F per minute.

2. Apply full vacuum. Continue raising of temperature at 6 F to 8 F per minute to 305 F.

3. Increase applied pressure to 90 psig, also maintaining full vacuum. Continue raising temperature at a rate of 6 F to 8 F per minute until 350 F ± 5 F is reached.

4. Hold at 350 F ± 5 F for 90 minutes.

5. Cool to 150 F under 90 psig and full vacuum before withdrawing from autoclave.

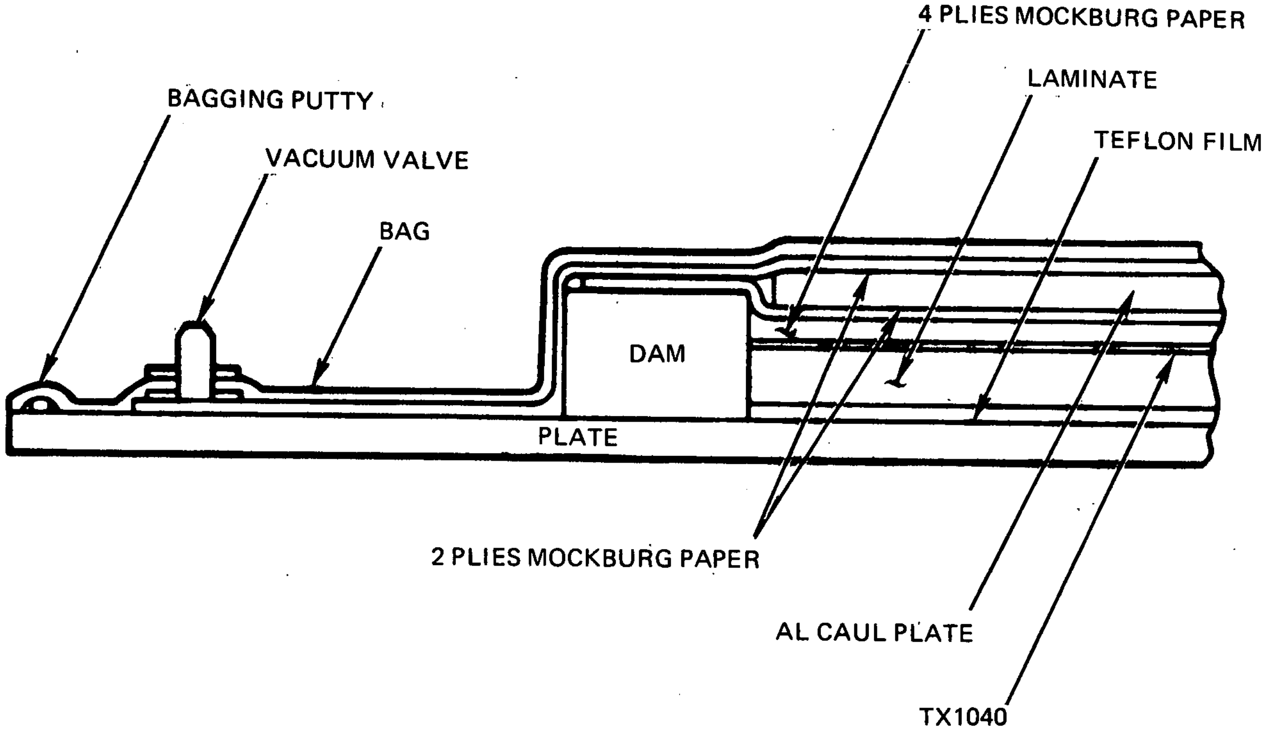

Figure A-5. Layup Diagram, Modified Gemon L Process

6. Post-cure between restraining steel sheets according to the following schedule:

   300 F for 14 hours
   350 F for 12 hours
   400 F for 36 hours
   450 F for 33 hours
   500 F for 14 hours

   Note: The heating rate between holding levels is not critical; 5 to 10 minutes is suggested to reach the next higher level.

7. Continue post-cure by raising temperature from 500 F to the specified maximum post-cure temperature at a rate of 1 F every 5 minutes. Hold at this temperature for 4 hours.

8. Cool to 150 F before removing restraining steel sheets.

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APPENDIX B. GEMON L/MODMOR II PROCUREMENT SPECIFICATION AND GEMON L POLYIMIDE RESIN IMPREGNATED GRAPHITE FIBER PREPREG PHYSICAL PROPERTY TESTING PROCEDURES

GEMON L/MODMOR II PROCUREMENT SPECIFICATION

1.0 Prepreg requirements

1.1 Fiber weight per unit area (gm/ft^2):

12.6 ± 1.3 for individual determinations
12.6 ± 1.0 for average of two samples

1.2 Volatiles (percent by weight): 6-10 (30 ± 2 minutes at 500 ± 10 F)

1.3 Resin solids (percent by weight): 46 ± 6 for individual determinations
46 ± 4 for average of two samples

1.4 Resin flow (percent by weight): 20 ± 5 (100 psi, 350 F, 15 minutes) (for reporting purposes only; not an absolute requirement)

1.5 Fiber collimation: the lay of the tow must not deviate from a straight line within the prepreg by more than 5 degrees.

1.6 Gaps (quadrants of 3 by 12 inches):

Allowable	Frequency	Percent of Quadrants
40 mils	0	0
30-40 mils	1	10
20-29 mils	2	25
20 mils	Unlimited	100

Successive gaps in line and separated by less than 1 inch will be considered as one gap.

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1.7 Fiber batch numbers and physical properties will be supplied for each batch of material (i. e., tensile strength, tensile modulus, and fiber density).

2.0 Laminate properties:

Uni-axial laminates prepared for verification of prepreg quality and moldability (cycles not to exceed 100 psi and 500 F) will exhibit the following properties (minimum) when normalized to a 60 ± 2 percent fiber volume content.

Property*	RT	500 F (after 1/2 hr @ 500 F)
Flexural strength (ksi)	175 ave	115 ave
	160 ind	105 ind
Flexural modulus (psi x 10^6)	15.0 ave	14.0 ave
	13.0 ind	12.0 ind
Short beam shear strength (ksi)	11.0 ave	6.0 ave
	10.0 ind	5.5 ind

*Flexural strength specimens will be tested using 32-to-1 span-to-thickness ratio. Short beam shear specimens will be tested using 4-to-1 span-to-thickness ratio.

3.0 Sheet will be a minimum of 3 inches wide

GEMON L POLYIMIDE RESIN IMPREGNATED GRAPHITE FIBER PREPREG PHYSICAL PROPERTY TESTING PROCEDURES

1.0 Graphite/Gemon L polyimide prepreg resin flow

1.1 Description

This test method consists of placing two cross-ply layers of polyimide/graphite prepreg between six layers of Mockburg paper bleeder with a piece of porous TX 1040 Teflon-coated glass cloth separating the prepreg from the bleeder paper. The total package is placed between aluminum caul plates in a preheated 350 F press and held for 15 minutes at 100 psi. The percent flow is based on the resin loss of the laminate.

1.2 Apparatus and material

The following items are required for performance of the test:

a. Hydraulic press with 400 F platens

b. Six squares of Mockburg paper, 3 by 3 inches, Grade CW1850, West Coast Paper Company

c. Two, squares 3 by 3 inches, of porous Teflon-coated fiberglass TX 1040, Pallflex Corporation

d. Two aluminum caul plates, 0.25 by 8 by 12 inches

e. Two pieces of Teflon sheet, 8 by 12 by 0.002 to 0.005 inches

f. Analytical balance

g. Desiccator

1.3 Test specimens

Individual test specimens are made up of two, 2-by-2-inch, crossplied (0 to 90 degrees) tapes.

1.4 Procedure

a. Weigh the prepreg, Mockburg paper, and TX 1040 fabric squares to the nearest 0.1 mg on an analytical balance.

b. Center the two layers of cross-plied (0 to 90 degrees orientation) prepreg tapes (1.3) between the two squares of TX 1040 fabric and the six layers of paper (three on top and three on bottom) and weigh to the nearest 0.1 mg.

c. Center the layered flow specimens on a Teflon-covered aluminum caul plate; cover with the remaining piece of Teflon and the second caul plate. Place in a 350 ± 10 F preheated platen press for 15 to 20 minutes at 100 psi (400 pounds platen force per test specimen). After 15 minutes in the press, remove the flow specimens from between the caul plates and allow to cool to room temperature.

d. Remove the cross-ply test specimen laminate from the TX 1040 fabric and paper. Weigh the TX 1040 fabric and paper to the nearest 0.1 mg.

1.5 Data reporting

Report the increase in weight of the TX 1040 fabric and Mockburg paper associated with each test specimen as resin loss or percent resin flow. Report to the nearest 0.1 percent.

1.6 Calculations

Calculate the increase in weight of the TX 1040 cloth and Mockburg paper according to the following equation:

$$\text{Percent flow} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

where

W_1 = weight of TX 1040 fabric and paper before pressing, grams

W_2 = weight of TX 1040 fabric and paper, and the two layers of prepreg, grams

W_3 = weight of TX 1040 fabric, paper, and resin after pressing, grams

Note: All materials must be stored in a desiccator between the above and all following operations.

2.0 Prepreg weight per unit area

2.1 Description

This test method consists of determining the unit area prepreg weight by accurately measuring the weight and dimensions of nominally 1.25 by 1.25 inch specimens and by performing the pertinent computation of weight divided by area. Since the same specimens are processed later for volatile and resin content and fiber weight per unit area determinations, the weighing steps are performed in the tare equipment required for these subsequent processing steps.

2.2 Apparatus and material

The following items are required for performance of the test:

a. Extraction thimbles, Fritted glass, medium E.C. 35 by 90 mm, Catalog No. 27743-120, Van Watters and Rogers Co., or equivalent

b. Analytical balance

2.3 Test specimen

Individual specimens will be cut to 1.25 ± 0.01 by 1.25 ± 0.01 inches.

2.4 Procedure

a. Obtain clean, dry extraction thimbles.

b. Determine the area of the individual specimens to an accuracy of 0.01 square inch.

c. Weigh each extraction thimble on an analytical balance to the nearest 0.1 mg. Place the test specimen in the extraction thimble and record the total weight to the nearest 0.1 mg.

2.5 Data reporting

Report the prepreg weight in grams per square foot.

2.6 Calculations

Calculate the prepreg weight per unit area according to the following equation:

$$\text{Prepreg weight/ft}^2 = \frac{W_1 - W_2}{A} \times 144$$

where

W_1 = weight of extraction thimble plus test specimen, grams

W_2 = weight of extraction thimble, grams

W_3 = area of prepreg specimen, square inches

E

3.0 Graphite/Gemon L polyimide prepreg volatile and resin content

3.1 Description

This test method consists of heating the carefully weighed prepreg weight per unit area specimen in a preheated over for 15 minutes and reweighing to determine total volatile content. Resin content is determined by digesting the same specimen in concentrated nitric acid for a minimum of three hours at 240 F. The residue is washed, dried, and weighed. The volatile and resin contents are determined from the weight loss after each operation.

3.2 Apparatus

a. Beaker, 2000 ml

b. Constant temperature hotplate

c. Nitric acid, 70 percent, reagent grade

d. Extraction thimbles, Fritted glass, medium E.C. 35 by 90 mm, Catalog No. 27743-120, Van Waters and Rogers Co., or equivalent

e. Acetone, reagent grade

f. Analytical balance

g. Drying oven capable of attainint 500 F

h. Pyrex vacuum filter flask, 500 ml

i. Vacuum rubber crucible holder

j. Vacuum source capable of at least 5 inches of mercury

k. Desiccator

3.3 Test specimen

Test specimens are taken from the previously measured prepreg weight per unit area measurement tests.

3.4 Procedure

a. Place the extraction thimbles containing the specimens in a preheated 500 ± 10 F air circulating oven for 28 to 32 minutes, cool to room temperature, and weigh to the nearest 0.1 mg. Store specimens in a desiccator between all operations.

b. Place the thimbles and contained specimens in a 2000 ml beaker. Add a minimum of 800 ml of nitric acid to the beaker. Place the beaker on a hotplate and maintain at a temperature of 240 + 10 F for a minimum of 3 hours, or until digestion is complete.

c. When digestion is complete, remove the extraction thimble from the beaker of acid and place in a rubber crucible holder on a vacuum filter flask. Initially, remove the acid, then subject the test specimen to a rinse with fresh acid, followed by a distilled water rinse and a final rinse of acetone.

d. Place the extraction thimbles and test specimens in a preheated drying oven at 215 ± 10 F for one hour, cool to room temperature in a desiccator, and weigh to the nearest 0.1 mg.

3.5 Data reporting

Report the resin and volatile contents, as percent by weight, to the nearest 0.1 percent.

3.6 Calculations

Calculate the volatile content and resin content according to the following equations:

$$\text{Volatile content, percent by weight} = \frac{W_1 - W_3}{W_1 - W_2} \times 100$$

$$\text{Resin solids content, percent by weight} = \frac{W_3 - W_4}{W_3 - W_2} \times 100$$

where

W_1 = weight of extraction thimble plus test specimen, grams, before volatile removal

W_2 = weight of extraction thimble, grams

W_3 = weight of extraction thimble plus specimen after volatile removal

W_4 = weight of extraction thimble plus specimen after extraction

4.0 Fiber weight per unit area

4.1 Description

This test method consists of weighing the remaining graphite fibers in the extraction thimble left from the resin content test, Paragraph 3. The sample is weighed to the nearest 0.1 mg, and the fiber weight per unit area is calculated.

4.2 Apparatus and material

The same apparatus employed in Paragraphs 1 through 3 is applicable.

4.3 Test specimen

The remaining fibers in the extraction thimble constitute the test specimen.

4.4 Procedure

Obtain the weight of fibers remaining in the extraction thimble from previous test, Paragraph 3, Resin Solids Content Test.

4.5 Data reporting

Report the fiber weight obtained as grams per square foot based on the original 1.25-by-1.25-inch sample tested in Paragraph 2.

4.6 Calculations

Calculate the fiber weight per square foot according to the following equation:

$$\text{Fiber weight per square foot} = \frac{W_5}{A} \times 144$$

where

W_5 = weight of fiber specimen after resin extraction, grams

A = original area of prepreg sample (Paragraph 2), square inches, $W_5 = W_4 - W_2$, (see Paragraph 3.6)

5.0 Tow count

5.1 Count the number of tow fiber bundles that make up the width of the prepreg.

5.2 Divide the total number of tow bundles by the width (inches). Record the number of strands per inch of width.

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APPENDIX C. PROCESS DESCRIPTION GRAPHITE – POLYIMIDE SIMULATED OV-10A CENTER WING SECTION

1.0 Scope

This process description outlines the materials and procedures to be employed in the fabrication of the graphite-polyimide simulated OV-10A Center Wing Section, Drawing MT-100017.

2.0 Applicable Documents and Materials

Productive Materials	Commercial Designation and/or Specification	Supplier
Glass Fabric, polyimide resin impregnated (prepreg)	35-512 & 35-503	Dupont
Honeycomb core-polyimide fiber paper impregnated with polyimide resin	HRH-327-3/16	Hexcel
Polyimide adhesive glass carrier supported	FM-34 (0.135 psf) (0.030 psf) MB0120-062	Bloomingdale
Polyimide primer	BR-34	Bloomingdale
Polyimide primer thinner	BR-34-2	Bloomingdale
Polyimide foam sheet	FM-29 Type II	Bloomingdale
Titanium sheet	6AL-4V MB-0170-052	Reactive Metals or Timet
Graphite fibers impregnated with polyimide resin	Gemon L/Modmor II	General Electric
Glass fabric	181	Commercial
Molded insert	MT-100013	NR/Columbus

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3.0 Requirements

3.1 Material Certification

Glass fabric/polyimide prepreg, graphite/fiber polyimide prepreg, and polyimide adhesive will be procured with supplier's certification of meeting the applicable specifications or procurement document requirements. Receiving Inspection tests will also be conducted upon material receipt to assure conformance to these specifications or requirements. In addition, recertification of these materials will be accomplished if evidence of improper storage or handling exists, or if the materials are within 14 days of storage period expiration date.

3.2 Materials Handling

3.2.1 Glass fabric/polyimide and graphite/polyimide prepregs will be stored in moisture-proof sealed bags at 40 F maximum.

3.2.2 FM-34 adhesive and FM-29 foam sheet will be stored in moisture-proof sealed bags at 0 F maximum.

3.2.3 BR-34 primer and BR34-2 primer thinner will be stored as received at 0 F maximum.

3.2.4 The handling of all flammable and dangerous liquids and chemicals will be in accordance with Code 1.

3.3 Mold Surface Preparation

3.3.1 Clean tool with MEK.

3.3.2 Apply parting agent to tool (Rulon Fluorocarbon Aerosol Spray or equivalent) and cure in accordance with supplier's recommendation.

3.4 Laminating Procedures

3.4.1 Thin Glass Fabric/Polyimide Details

3.4.1.1 Lay up 35-512 prepreg plies on the prepared tool in accordance with drawing requirements.

3.4.1.2 Cover with TX 1040 porous release film, followed by one ply of Mockburg paper bleeder for each two plies of glass fabric/polyimide prepreg.

3.4.1.3 Bag in conventional fashion and cure according to the following schedule:

a. Apply full vacuum.

b. Raise temperature to 250 ± 10 F and hold for a minimum of 8 hours.

c. Add 70 psig autoclave pressure.

d. Raise temperature to 310 ± 10 F and hold for 1.5 hours ± 10 minutes.

e. Raise temperature to 350 ± 10 F and hold for 1.5 hours ± 10 minutes.

f. Cool to 150 F under full vacuum and 70 psig autoclave pressure before removal from autoclave.

3.4.1.4 Post-cure according to the following schedule in restrained (on tool) condition:

a. Raise temperature to 350 ± 10 F and hold for 1 hour ± 10 minutes.

b. Raise temperature to 400 ± 10 F and hold for 1 hour ± 10 minutes.

c. Raise temperature to 450 ± 10 F and hold for 1 hour ± 10 minutes.

d. Raise temperature to 500 ± 10 F and hold for 1 hour ± 10 minutes.

e. Raise temperature to 550 ± 10 F and hold for 1 hour ± 10 minutes.

f. Raise temperature to 600 ± 10 F and hold for 2 hours ± 10 minutes.

g. Cool to 150 F in restrained condition before removal from the oven.

3.4.2 Thick Glass Fabric/Polyimide Details

3.4.2.1 Lay up 35-503 prepreg plies on the prepared aluminum layup plate in accordance with drawing requirements using the following sequence:

a. Follow every three plies of 35-503 prepreg with one ply of dry 181 glass fabric.

b. The final two plies shall be 35-503 prepreg.

3.4.2.2 Cover with TX 1040 porous release film, followed by 1/2-inch-thick 3/16-inch cell honeycomb core and three plies of Mockburg paper.

3.4.2.3 Bag in conventional fashion and cure according to the following schedule:

a. Apply full vacuum pressure.

b. Raise temperature to 350 ± 10 F and hold for a minimum of 8 hours in oven.

c. Cool to 150 F under full vacuum pressure before removal from oven.

d. Rebag without honeycomb core and continue cure in autoclave as follows:

(1) Apply full vacuum pressure and 90 psig positive pressure.

(2) Raise temperature to 350 ± 10 F and hold for 2 1/2 hours (minimum).

(3) Cool to 150 F under full vacuum and 90 psig positive pressure before removal from the autoclave.

3.4.2.4 Post-cure in fully restrained condition (in press) according to the following schedule:

a. Raise the temperature to 550 ± 10 F and hold for 4 hours.

b. Cool to 150 F while still under pressure before removing from the press.

3.4.3 Graphite/Polyimide Composite Details

Note: All of these details contain step lap titanium end fittings which are integrally bonded to the graphite/polyimide sections during the laminating operation with the aid of interposed FM-34 adhesive, 0.03 psf. For surface preparation and priming of the titanium end fittings, see Paragraph 3.5.1.

3.4.3.1 Prepare layup per drawing requirements and the arrangement shown in Figure C-1, utilizing the following number of Mockburg paper material plies:

Mockburg Paper	Spar Skins	Cover Panel Skins
Lower bleeder	2	3
Upper bleeder	4	4
Breather	2	4

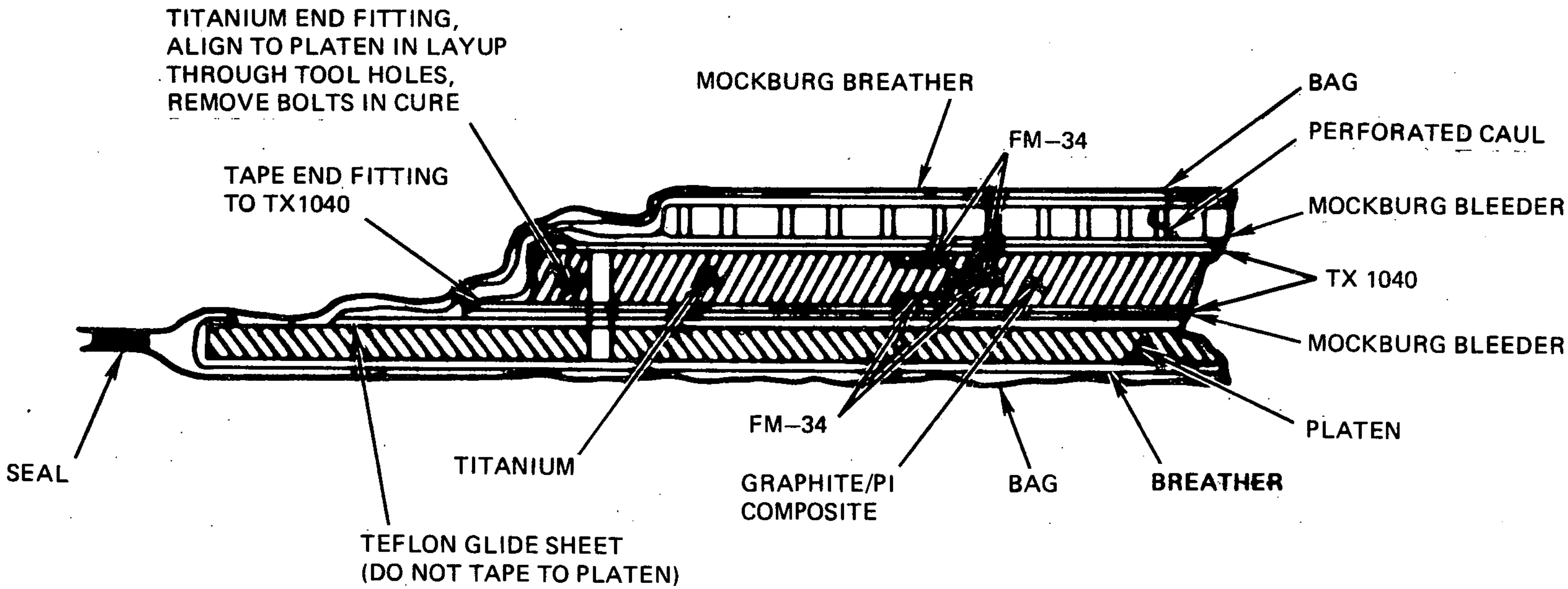

Figure C-1. Layup Schematic, Graphite Polyimide Skin Details

Note A: The layup operations will be accomplished in accordance with the following guidelines:

1. Layup the proper number of graphite prepreg plies for coordination with top face of middle step of titanium end fitting, i.e., 4 plies for spar skins and 7 plies for cover panel skins.

   In sequential layup of these plies, trim them individually to applicable tool marks on each end, utilizing temporarily interposed steel shims to prevent cutting through previous ply.

2. Add polyimide adhesive, FM-34, 0.03 psf, to the Gemon L/ Modmor II prepreg in the step areas.

3. Add titanium end pieces and add tool pins.

4. Add polyimide adhesive, FM-34, 0.03 psf, to the titanium end fittings in the step areas.

5. Repeat applicable operations until final plies are laid up.

Note B: The layup will be closed out longitudinally by Coroprene or aluminum bar dams.

3.4.3.2 Cure according to the following schedule:

a. Apply 90 psig positive pressure and full vacuum.

b. Raise temperature to 350 ± 5 F at a rate of 3 to 5 F per minute.

c. Hold at 350 ± 5 F for 2 hours ± 10 minutes.

d. Cool to 150 F before release of vacuum and positive pressure.

3.4.3.3 Post cure according to the following schedule in restrained condition:

a. Raise the temperature to 300 ± 10 F (no definite time).

b. Raise the temperature to 500 ± 10 F at the rate of 20 per hour and hold at 500 F for 18 hours ± 15 minutes.

c. Cool to 150 F before removing restraining members.

3.5 Surface Preparation for Bonding

3.5.1 Titanium Surfaces, Integral Bonding

3.5.1.1 Hydrohone

3.5.1.2 Immersion clean in accordance with Table 1.

3.5.1.3 Prime with BR-34 Primer, adjusted to 35 percent solids content with BR34-2 thinner. Priming is to take place preferably immediately after cleaning. (If this is not possible, cleaned details will be stored in sealed plastic bags.) Primer is to be applied so that the cured thickness is 0.0005 to 0.001 inch.

3.5.1.4 Cure the primer as follows:

a. Air dry for 30 to 40 minutes

b. Place the parts in an air circulating oven, raise the temperature to 240 ± 10 F, and hold for 30 to 40 minutes.

c. Raise the temperature to 400 ± 10 F and hold for 30 to 40 minutes.

3.5.1.5 Remove the parts from the oven and seal in a plastic bag until ready for bonding.

3.5.2 Titanium Surfaces, Secondary Bonding

3.5.2.1 Recondition bonding surfaces according to Table 2.

3.5.2.2 Reprime in accordance with Paragraph 3.5.1.3 and 3.5.1.4, except for adjusting primer solids content to result in a cured thickness of 0.001 to 0.002 inch.

3.5.3 Laminate Surfaces, Secondary Bonding

3.5.3.1 Glass fabric/polyimide laminates: lightly sand area to be bonded with 120 to 180 grit paper, followed by wiping with an MEK dampened rag.

3.5.3.2 Graphite/polyimide composite: lightly sand with Scotch-Brite Type "A". Remove dust by washing surface with MEK, followed by BR-34-2 thinner. Wipe dry with clean lint-free gauze.

3.6 Adhesive Bonding

Table 1. Titanium Alloys — Immersion Process

Step	Operation	Materials	Time (Minutes)	Temp (° F)	Remarks
1	Clean	MB0210-008 6-8 oz/gal	15 to 30	180 ± 10	See Note 1.
2	Rinse	Hot water	5 Minimum	150 ± 20	Inspect for water break
3	Immerse	Nitric acid 55 ± 3%; Ammonium biflucride, 3 ± 0.2%; balance water; all by weight	5 to 8	Room	
4	Rinse	Water	5 Minimum	Room	
5	Immerse	Tri-Sodium Phosphate, 5.0 ± 0.5%; Sodium Fluoride, 0.9 ± 0.2%; Hydrofluoric Acid, 1.6 ± 0.2%; balance water; all by weight	2 to 4	Room	Some titanium alloys may produce a smut in this solution. Remove loose smut only by spray rinsing; do not wipe. A surface coloration (gray) should be evident.
6	Rinse	Water	5 Minimum	Room	
7	Soak	Hot water	10 to 20	150 to 20	See Note 2.
8	Spray Rinse	MB0210-007 water (Ref. 3.8)	5 Minimum	150	Inspect for water break
9	Dry	Oven	30	230 ± 15	

NOTES: 1. MB0210-004 cleaner, 4 to 6 oz/gal at 145 ± 5 F, may be used as an alternative cleaner when hot water rinsing is not available
2. This step may be omitted if hot water rinsing is not available

3.6.1 Integral Bonding (Co-Cure)

Integral bonds between titanium end fittings and graphite/polyimide prepregs to be accomplished according to Paragraph 3.4.3.

3.6.2 Prefit, Component Details

Cured glass fabric/polyimide, graphite/polyimide details, and honeycomb core will be assembled in their correct respective positions on the intended bonding fixtures, with polyvinyl chloride (PVC) film substituted for the structural tape adhesive. The assembled details will be bagged and subjected to 13 psi minimum vacuum pressure. After disassembly, the simulated adhesive film will be examined for thickness and for impressions received. Where glueline thickness beyond tolerable limits for one ply of FM-34, i.e., 0.015 inch, is indicated, local corrections will be made by (a) appropriate local machining, (b) provision for local increase in number of adhesive plies, or (3) a combination of both.

3.6.3 Prefit, Spar-Rib Sub-Assembly and Final Assembly Components

These prefit operations will be conducted according to Paragraph 3.6.2 with the following exceptions:

a. Use of Verifilm FM-641 (Bloomingdale) in lieu of PVC film

b. Employment of the total intended pressure/temperature/time cure cycle

3.6.4 Component Honeycomb Sandwich Bonding

3.6.4.1 HRH-327-3/16 core sections will be spliced by overlap/interlock, or by butt-joining with interposed FM-29 Type II polyimide foam sheet. The latter will be cured simultaneously with subsequent bond cures.

3.6.4.2 The core will be roller coated on both sides with BR-34 primer to result in a total pickup of 27 to 32 gm/ft^2, evenly distributed between the two surfaces, after drying for 30 to 40 minutes at 250 F.

Note: Shrinkage of net dimensioned core blanks during drying can be avoided by use of an appropriate shop aid, keeping the core in a taut, expanded condition during drying. A metal sheet with protruding nails spaced on 2-3 inch centers in rows of 10 inches is satisfactory.

Table 2. Pre-Bond Surface Preparation,
Titanium Alloys - Manual Process

Step	Operation	Materials	Remarks
1	Remove cured primer	No. 320 grit aluminum oxide paper	Sand manually or with vibrator-sander until all traces of primer removed.
2	Remove primer residue	Tap water soaked cheesecloth followed by MEK dampened cheesecloth	Swab and wipe surfaces with cheesecloth, inspect, repeat Step 1 as required.
3	Mask off bond areas	Aluminum foil tape, Scotch No. 425, or lead foil tape, Scotch No. 420	Allow about 1/2 inch excess beyond bond area. Carefully mask off and seal areas next to bond lines and composite surfaces. Seal tape on Ti surface 1/4 to 3/8 inch back from any point or composite surface.
4	Abrasively clean bond surfaces	No. 320 grit aluminum oxide paper	Thoroughly abrade bonding surfaces[1].
5	Clean bond areas	Clean cheesecloth dampened with tap water and squeeze bottle with tap water.	Wipe bond area clean with tap water dampened cheesecloth.
6	Chemically clean bond surfaces	Pasa Jell 107 paste Note: Strong acid. Do not breathe vapor, get in eyes, on skin, or clothing. Do not allow contact with combustibles. Use with approved vent.	Dispense a quantity of Pasa Jell 107 paste on to a bond area using an acid brush. Cross brush the paste every 3 to 5 minutes and add new material as required to keep fresh acid in contact with the titanium.

F

Table 2. (Continued)

Step	Operation	Materials	Remarks
6 Cont			Maintain operation for 20-25 minutes. Use clean cloth gloves for all subsequent operations.
7	Rinse bond surfaces	Clean cheesecloth dampened with distilled water and squeeze bottle with distilled water.	Wipe bond area free of Pasa Jell 107 paste with cheesecloth. Wipe clean with water-dampened cheesecloth. Flood area with distilled water. Insure that no liquid residue contacts composite surfaces or bond joints.
8	Water break test	Distilled water squeeze bottle	Examine parts rinsed with clean, running distilled water for continuity of the water film. Spray assembly with squeeze bottle of distilled water while bonding surface is in a vertical position. Formation of water droplets or discontinuity of the water film indicates presence of oily or greasy residues; reprocess part.
9	Dry bond area	Heat gun or drying oven	Wipe surface dry with clean cheesecloth. Cheesecloth must not show evidence of residue. Force dry the bond area using a heat gun or air circulating oven at approximately 200 F for 30 minutes.

Table 2. (Continued)

Step	Operation	Materials	Remarks
10	Test pH of titanium bonding surface	Distilled water in squeeze bottle Hydrion paper	After rinsing and drying, select a representative area of the bonding surface and wet this area with a few drops of distilled water. Test the wet area with Hydrion pH indicating paper. A pH of less than 5.0 indicates acidic residues; rerinse part. Force dry the pH test area in accordance with Step 9.
11	Removal of masking tape	None	Remove masking tape (see Step 4).
12	Recleaning of masked area	Clean cheesecloth dampened with MEK	Wipe area from which masking tape has been stripped with MEK dampened cheesecloth.[2]

[1] Be careful not to abrade adjacent composite members in this operation.

[2] Be careful to confine this operation essentially to previously masked area only.

3.6.4.3 Faying details shall be assembled with interposed FM-34, 0.135 psf adhesive, bagged, and cured in accordance with the following schedule:

a. Raise part temperature to 120 F.

b. Apply full vacuum and hold throughout the entire cure cycle.

c. Raise the part temperature from 120 F to 270 ± 10 F in 42 minutes ± 17 minutes.

d. Raise the part temperature from 270 F to 342 ± 12 F in 65 ±25 minutes, and apply 35 to 55 psig autoclave at 300F + 30 F.

e. Hold at 342 ± 10 F for 2 hours ± 10 minutes.

f. Allow assembly to cool down to 120 F before releasing pressure.

3.6.4.4 Post-cure in restrained condition (on tool) according to the following schedule:

a. Raise temperature to 400 ± 10 F in one hour ± 15 minutes and hold for 3 hours.

b. Raise temperature to 500 ± 10 F in one hour ± 15 minutes and hold for 4 hours.

c. Cool to 150 F in restrained condition before removal of restraining members.

3.6.4.5 Core edges remaining exposed after final assembly cure will be filled with YM-25, NR/SD proprietary compound. This compound is cured during final assembly post-cure, (Paragraph 3.6.5.5).

3.6.5 Secondary Continuous Surface Bonding, Spar-to-Rib Subassembly, and Final Assembly

3.6.5.1 Assemble components with the aid of the appropriate mechanical assembly aid fasteners and utilizing FM-34, 0.135 psf adhesive at the bond line interfaces. One layer of adhesive will be used in the subassembly and two layers in the final assembly joints, with local corrections as found appropriate from the prefit impression checks (Paragraph 3.6.3).

3.6.5.2 Install process control thermocouples and bag in silicone rubber bag; apply 25 inches of Hg minimum vacuum pressure.

3.6.5.3 Position in bonding fixture, with in-line process control coupons in their appropriate locations under the pressure application tool members; apply 5 psi positive pressure.

3.6.5.4 Cure according to the following schedule:

a. Adjust, if necessary, to full vacuum and hold throughout the cure cycle.

b. Close the autoclave.

c. Raise the part temperature to 300 ± 30 F, 2 to 4 F per minute.

   Raise the positive (tool-applied) pressure from 5 psig to 55 psig and hold for the remainder of the cure cycle.

d. Raise the temperature to 350 ± 10 F, 2 to 4 per minute and hold for two hours ± 10 minutes.

e. Cool to 150 F under full vacuum and positive pressure.

3.6.5.5 Post-cure according to the following schedule:

a. Raise the temperature of the assembly to 200 ± 10 F and hold for one hour; raise the temperature to 300 ± 10 F and hold for one hour; raise the temperature to 400 F and hold for two hours.

b. Raise the temperature of the assembly to 500 F and hold for two hours (temperature rise rate not critical).

3.7 Article Assembly

Add door, bearings, retainers, and peel fasteners in accordance with Drawing MT1-00017; perform final trim and cleanup operations.

4.0 Quality Assurance

Article quality will be monitored by receiving inspection of incoming productive materials, by fabrication and test of process control coupons, and by dimensional inspection of fabricated details, components, subassemblies, and assemblies to drawing callouts.

4.1 Pertinent acceptance criteria guidelines for process control coupons are delineated below.

a. Graphite/polyimide composite

	Minimum Average	Minimum Individual Value
RT flexural strength, ksi[1]	175	160
500 F flexural strenght, ksi	115	105
RT flexural modulus, msi[1]	15.0	14.0
500 F flexural modulus, msi[1]	13.0	12.0

b. Glass fabric/polyimide laminate

RT flexural strength[1]	72 ksi min
500 F flexural strength	45 ksi min
RT flexural modulus[1]	2.6 msi min
500 F modulus[1]	2.1 msi min

c. FM-34 bonds

RT tensile shear strength[1, 2]	2500 psi min
500 F tensile shear strength[2]	1200 psi min
500 F flatwise tensile strength[3]	500 psi min

[1]Optional test

[2]Ti-to-Ti preferably, 1/2-in. O.L.

[3]0.020 Al facings to 3/16-0.002 or 0.003 Al core

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APPENDIX D. DESIGN DRAWINGS

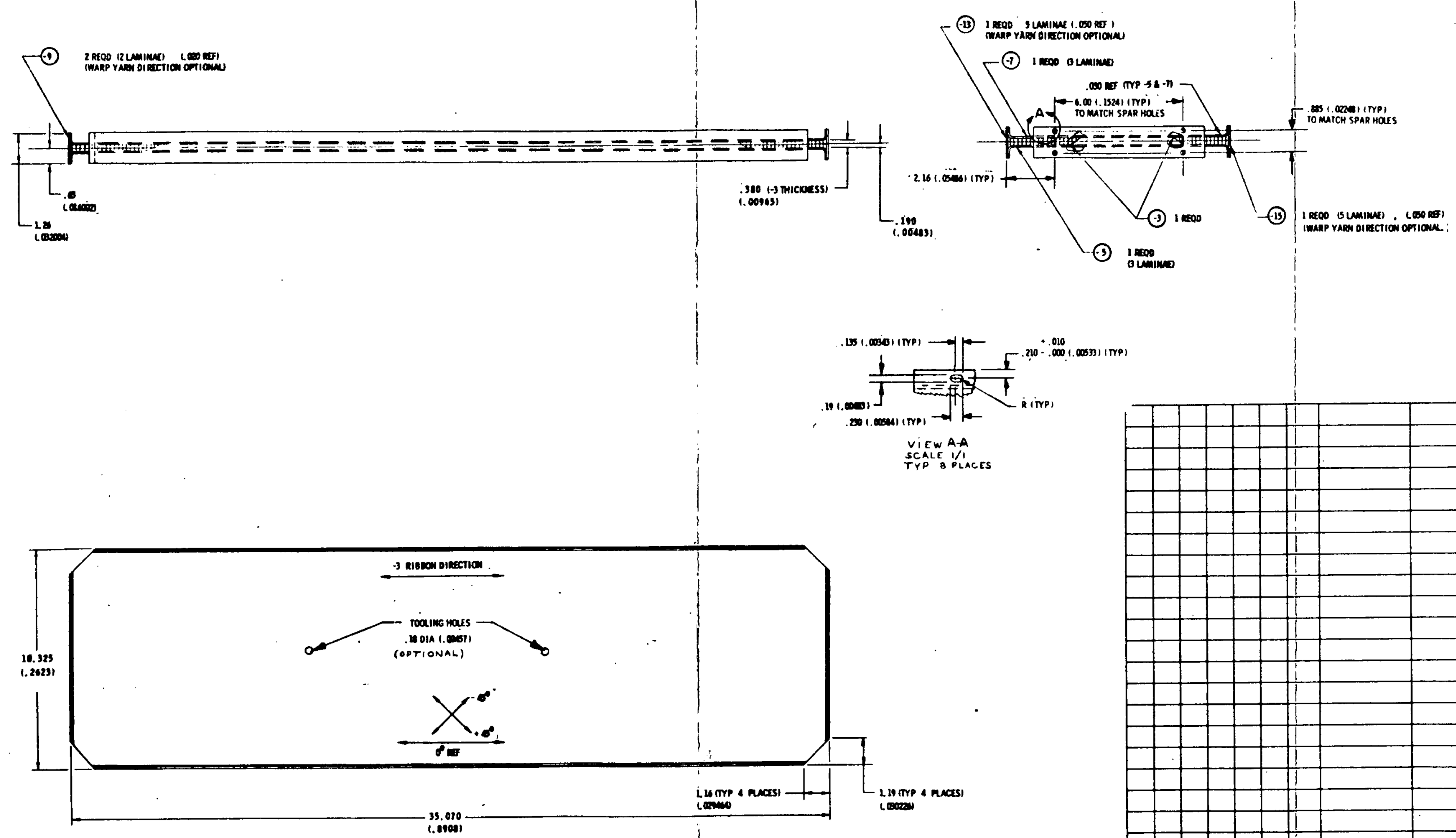
This appendix contains the final design drawings for the graphite/polyimide center wing box beam. These drawings are listed below:

Figure Number	Drawing Number	Title
D-1	MT-100011	Rib-Wing Nonmetallic Center Box Assemb Assembly of Test Specimen
D-2	MT-100012	Spar-Wing Nonmetallic Center Box Assembly of Test Specimen
D-3	MT-100013	Insert-Wind Nonmetallic Center Box Test Specimen
D-4	MT-100014	Panel-Wing Nonmetallic Center Box Upper Skin Assembly of Test Specimen
D-5	MT-100015	Door-Wing Nonmetallic Center Box Assembly of Test Specimen
D-6	MT-100016	Panel-Wing Nonmetallic Center Box Lower Skirt Assembly of Test Specimen
D-7	MT-100017	Beam-Wing Nonmetallic Center Box Assembly of Test Specimen

FOLDOUT FRAME 1

FOLDOUT FRAME 2

ZONE	LTR	DESCRIPTION	DATE	APPROVED
3	A	ADDED ASSEMBLY AID HOLES FOR ASSY TO SPARS. ADDED TOOLING HOLES		



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11. FILL EXPOSED CORE EDGES PER SK [illegible].
10. FACE SHEETS MUST BE BALANCED ACROSS CORE.
9. PARENTHETICAL DIMENSIONS ARE IN SI UNITS.
(2) 8. MAY BE PURCHASED FROM E. I. DUPONT, WILMINGTON, DEL.
(1) 7. MAY BE PURCHASED FROM HEXCEL PRODUCTS, INC., DUBLIN, CALIF.
6. WARP YARN DIRECTION FOR -5 & -7 TO BE ± 45° ALTERNATED.
5. BEND RADII .06.
4. 125 ALL MACHINED SURFACES.
3. MACHINE PER NR SPEC LA [illegible].
2. IDENTIFY PER NR SPEC LA 0104-003 EXCEPT DO NOT METAL IMPRESSION STAMP.
1. FABRICATE PER NR SK [illegible].

NOTES: UNLESS OTHERWISE NOTED

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Figure D-1

QTY REQD	QTY REQD	QTY REQD	QTY REQD	QTY REQD	QTY REQD	CODE IDENT	PART OR IDENTIFYING NUMBER	NOMENCLATURE OR DESCRIPTION	MATERIAL	DATA SPECIFICATIONS SIZES, NOTES, SUPPLIERS	ZONE
					1		-15	CAP	GLASS FABRIC PREPREG	(2) 35-512 .010 X .15 SQ. FT.	4
					1		-13	CAP	GLASS FABRIC PREPREG	(2) 35-512 .010 X .15 SQ. FT.	4
					1		-9	CAP	GLASS FABRIC PREPREG	(2) 35-512 .010 X .15 SQ. FT.	8
					1		-7	CHANNEL	GLASS FABRIC PREPREG	(2) 35-512 .010 X 8.25 SQ. FT.	4
					1		-5	CHANNEL	GLASS FABRIC PREPREG	(2) 35-512 .010 X 8.25 SQ. FT.	4
					1		-3	CORE	HONEYCOMB CORE	(1) HRH 327 -3/16-6.0 .380 X 10.31 X 35.00	3
					-11		MT-100011	ASSY OF			

PARTS LIST

-11	2		TEST		
REQD PER END ITEM	QTY	NEXT ASSY	USED ON	END ITEM NO.	THRU
		APPLICATION		EFFECTIVITY	

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES
TOLERANCES ON DECIMALS: .XX = ± .03; .XXX = ± .010. ANGLES ± 0° 30'
HOLES NOTED "DRILL"

DR BY GEC SCOTT 5-9-71
CHK BY
APPROVED BY

SPACE DIVISION
NORTH AMERICAN ROCKWELL

RIB-WING NON-METALLIC CENTER BOX ASSY OF TEST SPECIMEN

SIZE E | CODE IDENT NO 03953 | DRAWING NO. MT-100011

MT-100011

- 175 -

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FOLDOUT FRAME 1

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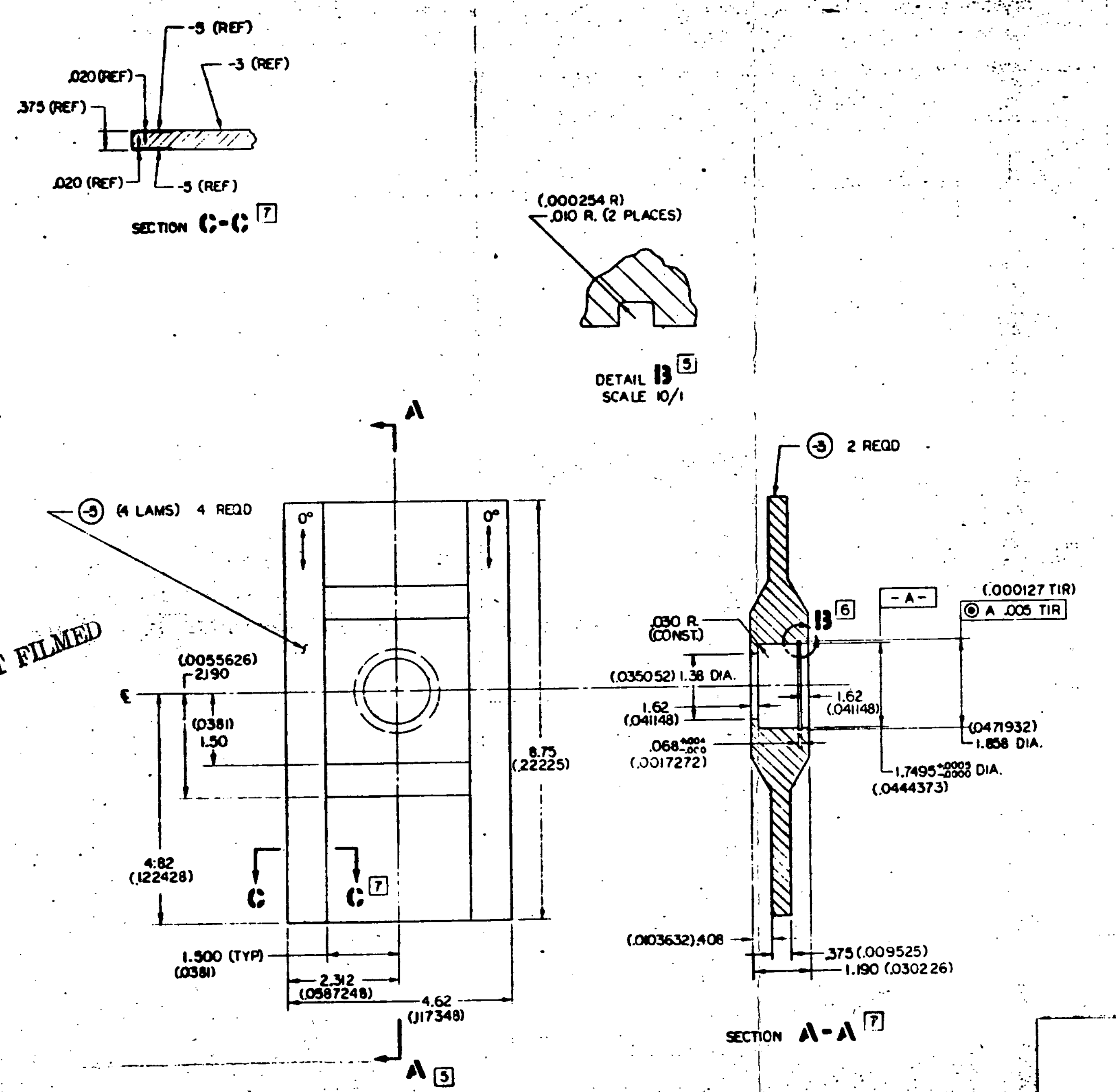

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(2) 5- MAY BE PURCHASED FROM WHITTAKER R&D SAN DIEGO, CALIF.

(1) 4- MAY BE PURCHASED FROM FIBERITE CORP., WINONA, WIS.

3- FABRICATE PER NR SPEC ST0605HA0015

2- IDENTIFY PER NR SPEC LA0104-003 EXCEPT DO NOT METAL IMPRESSION STAMP

1- PARENTHETICAL DIMENSIONS ARE IN SI UNITS

NOTES: UNLESS OTHERWISE NOTED

QTY REQD	QTY REQD	QTY REQD	QTY REQD	QTY REQD	QTY REQD	CODE IDENT	PART OR IDENTIFYING NUMBER	NOMENCLATURE OR DESCRIPTION	MATERIAL	DATA SPECIFICATIONS SIZES, NOTES, SUPPLIERS	ZONE
					4		-5	STIFFENER	BORON POLYIMIDE PREPREG	.197 SQ.FT. x .0052THK (2)	
					1		-3	INSERT	HFE-9020 POLYIMIDE MOLD COMPOUND	(1)	
					-11		MT-100013	ASSY OF			

PARTS LIST

-11	4		TEST		
ITEM	QTY	NEXT ASSY	USED ON	FND ITEM NO	
REQD PER END ITEM		APPLICATION		EFFECTIVITY	

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES
TOLERANCES ON DECIMALS .XX = ± .03, .XXX = ± .010; ANGLES ± 0° 30'
HOLES NOTED "DRILL"

DR BY
CHK BY
APPROVED BY

SPACE DIVISION
NORTH AMERICAN ROCKWELL

INSERT-WING NON-METALIC CENTER BOX (TEST SPECIMEN)

SIZE E | CODE IDENT NO 03953 | DRAWING NO. MT-100013

Figure D-3

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-179-

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Space Division
North American Rockwell

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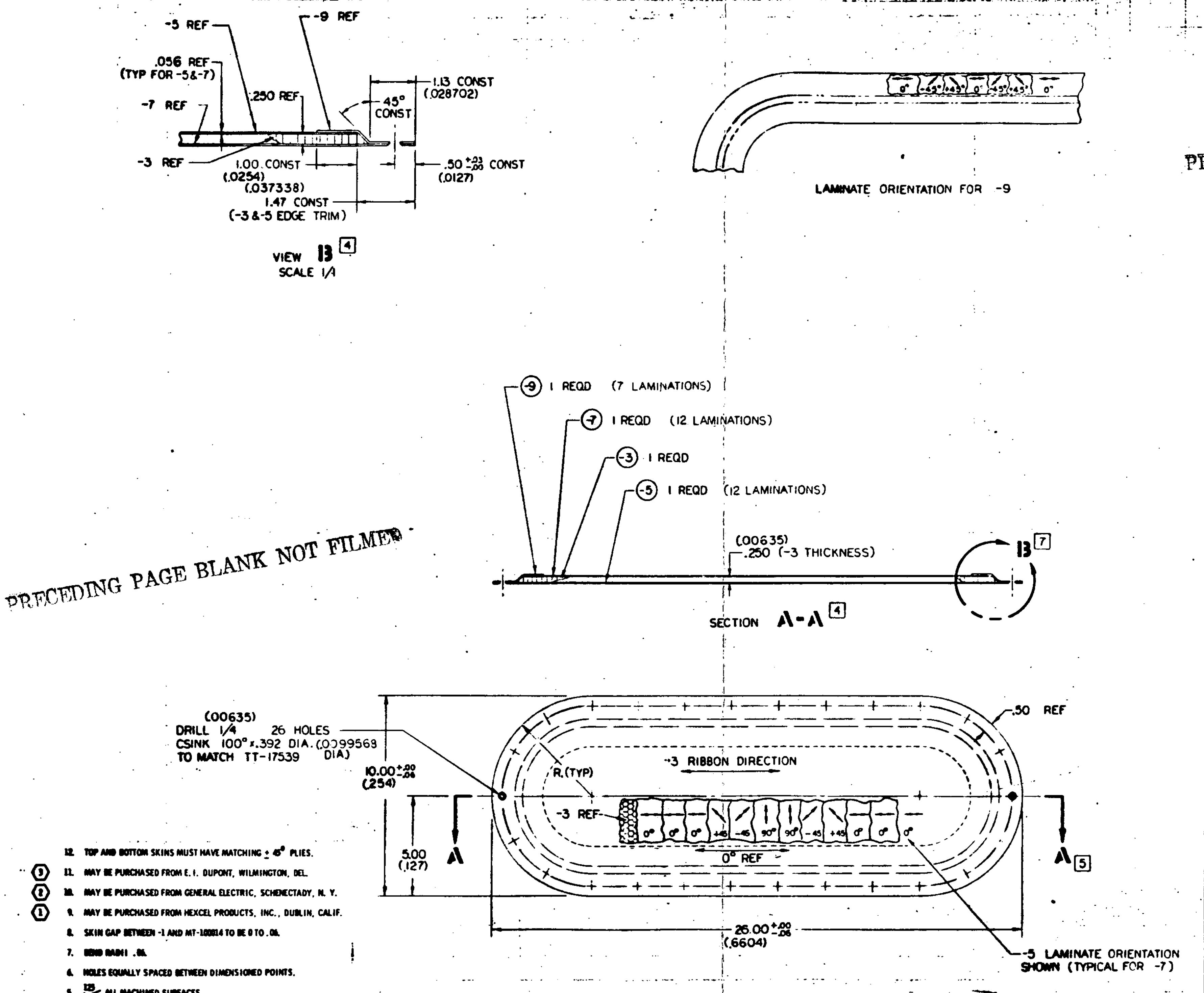

12. TOP AND BOTTOM SKINS MUST HAVE MATCHING ± 45° PLIES.

(3) 11. MAY BE PURCHASED FROM E. I. DUPONT, WILMINGTON, DEL.

(2) 10. MAY BE PURCHASED FROM GENERAL ELECTRIC, SCHENECTADY, N. Y.

(1) 9. MAY BE PURCHASED FROM HEXCEL PRODUCTS, INC., DUBLIN, CALIF.

8. SKIN GAP BETWEEN -1 AND MT-100014 TO BE 0 TO .06.

7. BEND RADII .06.

6. HOLES EQUALLY SPACED BETWEEN DIMENSIONED POINTS.

5. 125 ALL MACHINED SURFACES.

4. IDENTIFY PER NR SPEC LA 0104-003 EXCEPT DO NOT METAL IMPRESSION STAMP.

3. MACHINE PER NR SPEC MA 0103-005.

2. PARENTHETICAL DIMENSIONS ARE IN SI UNITS.

1. FABRICATE PER NR SPEC SK 15667.

NOTES: UNLESS OTHERWISE NOTED

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QTY REQD	QTY REQD	QTY REQD	QTY REQD	QTY REQD	QTY REQD	CODE IDENT	PART OR IDENTIFYING NUMBER	NOMENCLATURE OR DESCRIPTION	MATERIAL	DATA: SPECIFICATIONS SIZES, NOTES, SUPPLIERS	ZONE
					1		-9	CLOSEOUT	35-512 GLASS FABRIC PREPREG	8.10 SQ. FT. x .010 THK (3)	
					1		-7	FACE	PREPREG	MODMORE II / GEMON L 13.50 SQ. FT. X .0056 THK (2)	
					1		-5	FACE	PREPREG	MODMORE II / GEMON L 13.50 SQ. FT. X .0056 THK (2)	
					1		-3	CORE	HONEYCOMB CORE	HRH327-3/16 - 6.0 7.75 x 23.75 x .250 THK (1)	
					-11		MT - 100015	ASSY			

PARTS LIST

UNLESS OTHERWISE SPECIFIED DIMENSIONS ARE IN INCHES. TOLERANCES ON: DECIMALS ANGLES HOLES

-11 | 1 | TEST

NEXT ASSY | USED ON | APPLICATION | END ITEM NO. | EFFECTIVITY

DR BY | CHK BY | APPROVED BY

SPACE DIVISION
NORTH AMERICAN ROCKWELL

DOOR-WING NON-METALLIC CENTER BOX ASSY OF (TEST SPECIMEN)

SIZE E | CODE IDENT NO 03953 | DRAWING NO MT-100015

Figure D-5

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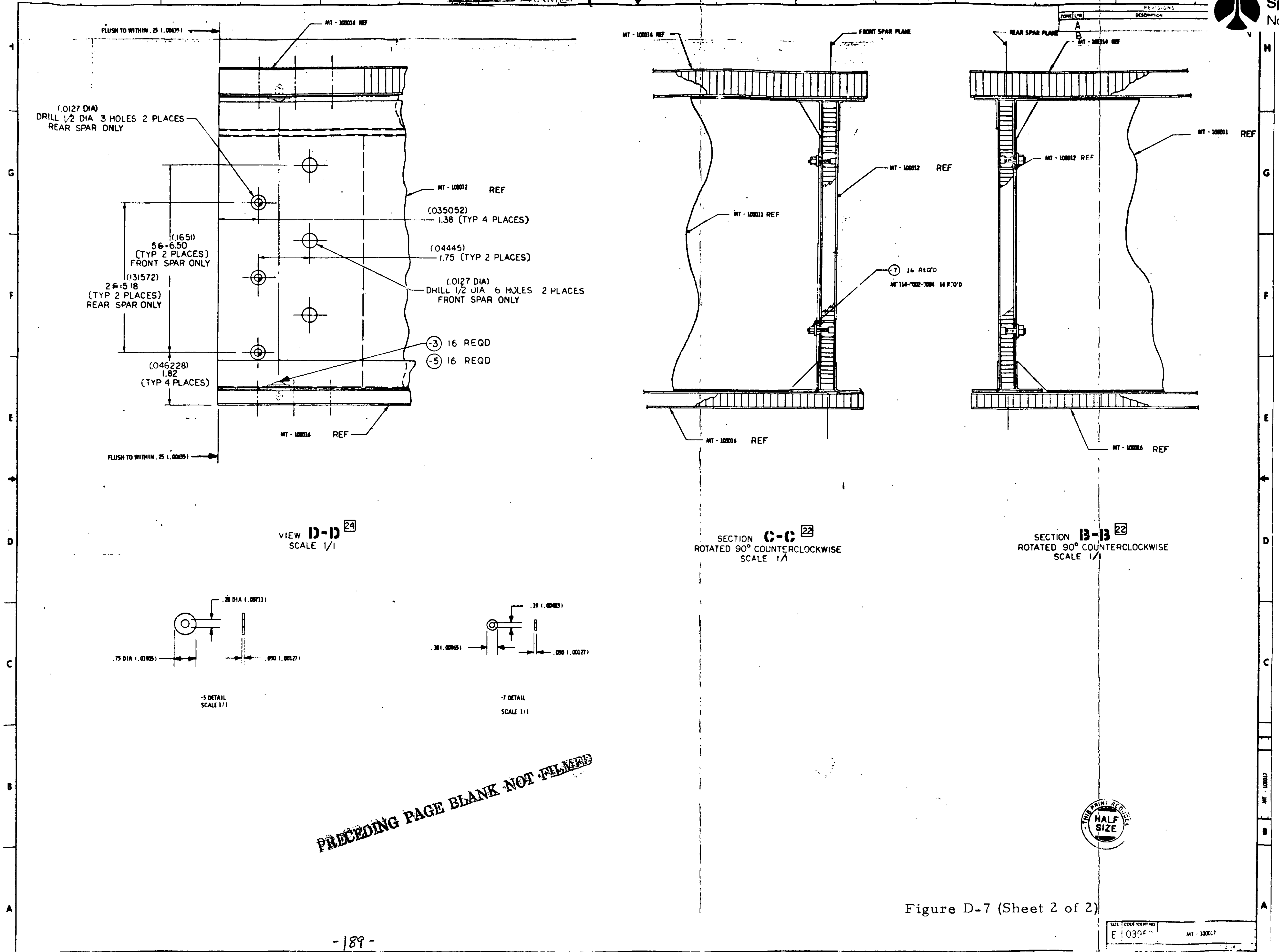

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Figure D-7 (Sheet 2 of 2)

APPENDIX E. TEST REPORT FORMAT

The following page is an example of an incoming material test report on the graphite/polyimide box beam program.

Space Division
North American Rockwell

SPACE DIVISION
NORTH AMERICAN ROCKWELL CORPORATION

TEST REPORT

DATE REC'D. IN LAB 2-12-71

L/R NO. T1-799

P.O. NO. MIE-3GDV-635-180H

MATERIAL Gemon L Graphite Prepreg

SPECIFICATION

SOURCE General Electric Company

QUANTITY 9 Sheets (To Lab/Received)

LAB FILE NO.

Test	Required	Sheet No.	Test Results
Resin Solids (2) %	46 + 6 Ind. 46 + 4 avg.	011371F	43.7, 40.3 avg. = 47.0
		012571D	42.6, 43.4 avg. = 43.0
		012271E	44.6, 48.0 avg. = 46.3
		012571A	44.3, 52.6 avg. = 48.4
Volatiles %	6 - 10%	011371F	9.4
		012571D	5.7
		012271E	6.4
		012571A	6.4
Fiber wt. (2) per unit area (g/sq. ft.)	12.6 ± 1.3 Ind. 12.6 ± 1.0 avg.	011471F	11.45, 12.05 avg. = 11.75
		012571D *	11.60, 10.92 avg. = 11.26
		012271E	13.15, 12.33 avg. = 12.74
		012571A	11.17, 12.50 avg. = 11.83
Prepreg wt. per unit area (g/sq. ft.)	N/A	011371F	24.45, 20.12 avg. = 22.28
		012571D	21.43, 22.04 avg. = 21.73
		012271E	24.33, 27.08 avg. = 25.70
		012571A	22.18, 30.77 avg. = 26.47
Tow collimation and gaps (1)	Parallel WITHIN + 5°	011371F	Conforms/conforms
		012571D	Conforms/conforms
		012271E	Conforms/conforms
		012571A	Conforms/conforms

Sheet No. 011371F also represents Sheet No. 011471C
Sheet No. 012571D also represents Sheet No. 012571C
Sheet Nos. 012571A and 012271E also represent Sheet Nos. 012271B, 012671A, and 012271B

Resin solids and fiber weight per unit area corrected for graphite fiber loss during acid digestion by factor of 1.01

*Not to requirements

CONFORMS TO REQUIREMENTS LISTED ☐ DOES NOT CONFORM TO REQMTS LISTED ☐

SIGNATURES

TESTED BY ______ DATE ______ SUPERVISOR ______ DATE ______

COMMENTS (1) See requirements in IL 191-201-71-022
(2) Acid digestion for these tests was performed at 215F for 10 hours

FORM 883-C-1 REV. 8-68

APPENDIX F. TYPICAL SHOP OPERATING PROCEDURE

This appendix presents a typical shop operating procedure as used in the Graphite/Polyimide Box Beam Program.

SK-015659 - Manufacturing Procedure for Spar Fabrication, Polyimide Box Beam

1.0 Materials

Type	Specification	Supplier
Glass fabric, polyimide resin impregnated (pre-preg)	35-512 & 35-503	Dupont
Honeycomb core-polyimide fiber fiber paper impregnated with polyimide resin	HRH-327-3/16	Hexcel
Polyimide adhesive glass carrier supported	FM-34 (0.135 psf) (0.030 psf)	Bloomingdale
Polyimide primer	BR-34	Bloomingdale
Polyimide primer thinner	BR-34-2	Bloomingdale
Polyimide foam sheet	FM-29, Type II	Bloomingdale
Titanium sheet	6AL-4V MB-0170-052	Reactive Metals or Timet
Graphite fibers impregnated with polyimide resin	GemonL/Modmor II	General Electric
Glass fabric	181	Commercial

2.0 Detail and/or subassembly drawings included in spar assembly

Name	Part or Identification Number	Supplier
Molded insert	MT-100013	NR/Columbus

3.0 General notes

3.1 Same as SK-015653 (2.1), except add Gemon L/Modmor II

3.2 Same as SK-015653 (2.2 through 2.4)

3.3 Reference sketches

a. Same as SK1015653 (2.5a)

b. SK-015665

Application of bleeders, release fabrics, thermocouple locations, vacuum outlets, and vacuum bag sealing procedures. For layup of Germon L/Modmor II face sheets.

3.4 Tooling required to fabricate the spars

a. SK-015657

Layup tool for spar angles

b. SK-015654

Layup tool for face sheets

c. SK-015655

Application of template to locate insert and access holes

d. SK-015656

Bonding jig for face sheet to core and inserts

e. SK-015658

Tooling bars to aid in bonding angles to subassembly

4.0 Tool preparation

4.1 Same as SK-015653 (e.1)

4.2 Same as SK-015653 (3.2)

5.0 Titanium edging

5.1 Cut material to rough size (7 1/2 by 21 1/2 inches).

5.2 Chem mill in accordance with drawing, check dimensions, and record.

5.3 Shear width to 7.00 inches.

5.4 Add tool holes (use SK-015654 for hole location).

5.5 Clean in accordance with MA0110-024, Table VI.

5.6 Prime with BR-34 primer (35 percent solids) immediately after parts are cleaned.

If this is not possible, place the cleaned parts in a sealed plastic bag. Apply the primer to a thickness of 0.0005 to 0.001 inch.

5.7 Cure the primer as follows:

a. Place the parts in an air-circulating oven; raise the temperature to 240 F ± 10 F and hold for 30 to 40 minutes.

b. Raise the temperature to 400 F ± 10 F and hold for 30 to 40 minutes.

5.8 Remove the parts from the oven and seal in a plastic container until ready for bonding.

6.0 Layup (face sheets)

6.1 Remove the Gemon L/Modmor II and polyimide adhesive (FM-34 0.030 psf) from refrigeration and record sheet number, lot number, batch number, and verification date of the Gemon L/Modmor II, and the roll number, batch number, and verification date on the FM-34 adhesive. Note: Allow the materi al to come to room temperature before removal from sealed container.

6.2 Apply bottom bleeder cloth (2 plies).

6.2.3 Cut the Gemon L/Modmor II material to the required 6-inch width and lay up one 45 degree ply on Tool SK-015654. (Refer to SK-015670 for ply orientation.)

6.4. Using the line layout on Tool SK-015654, trim both ends.

6.5 Repeat 6.3 and 6.4 until 4 plies are laid up, except that, prior to trimming ends, a steel shim will be added between plies to prevent cutting through the previous ply.

6.6 Add polyimide adhesive (FM-34, 0.035 psf) to the Gemon L/Modmor II in the step areas.

6.7 Add titanium end pieces and add tool pins.

6.8 Add polyimide adhesive (FM-34, 0.035 psf) to the titanium in the step areas.

6.9 Repeat 6.3 and 6.4 until two plies are laid up, except that, prior to trimming ends, a steel shim will be added between the titanium and graphite polyimide ply. Add thermocouples.

6.10 Lay up lap shear and flexure test coupons, and add thermocouples (lap shear 0.063 inch titanium bond with 2 plies of 0.030 psf FM-34 — inadvertently one ply of 0.135 psf was used). (Flex 13 plies Gemon L/ Modmor No. 4 x 4.)

6.11 Apply parting fabric to skins, add 4 plies of bleeder cloth and 2 plies of bleather cloth, add vacuum bag material. Note: Include skins and coupons in one basic tool and vacuum bag. (Refer to sketch No. SK-015665 for actual procedure.)

6.12 Add static vacuum gauge and vacuum line connections.

Check for leaks by applying 26 inches Hg vacuum pressure and shutting off vacuum source, then checking the static gauge for rate of drop; if the drop is greater than one inch of Hg per minute, the bag is improperly sealed and will be repaired accordingly.

7.0 Curing (facing sheets)

7.1 Place entire bagged unit into the autoclave.

7.2 Connect vacuum lines and thermocouple connections.

7.3 Recheck for vacuum loss as in 6.12 above.

7.4 Prepare cure card (Form 2972Y) and record serial number in laboratory notebook.

7.5 Secure autoclave, and cure skins according to the following schedules:

a. Apply 90 psig positive pressure, and full vacuum.

b. Raise temperature to 350 F ± 5 F at a rate of 3 to 5 F per minute.

c. Hold at 350 F ± 5 F for 2 hours ± 10 minutes.

d. Cool to 150 F before release of vacuum and positive pressure.

7.6 Disconnect thermocouple leads, remove bag and bleeder cloth, etc.

7.7 Visually inspect.

8.0 Postcure (face sheets)

8.1 Position face sheets and test coupons on SK-015654, add thermocouples, and hold in a restrained condition.

8.2 Place tool and skins in the oven and connect thermocouple leads.

8.3 Prepare cure card (Form 2972Y) and record serial number in laboratory notebook.

8.4 Secure oven and post cure face sheets and test coupons according to the following schedule:

a. Raise the temperature to 300 F ± 10 F (no definite time).

b. Raise the temperature to 500 F ± 10 F at the rate of 20 F per hour and hold at 500 F for 18 hours ± 15 minutes.

c. Cool to 150 F before removing restraining members.

8.5 Disconnect thermocouple leads, remove vacuum bag and bleeder cloth, etc.

8.6 Visually inspect.

8.7 Submit test coupons on Form 2906-C to Process Control Laboratory, D/098, for test.

8.8 Verify process control lab report.

8.9 Using template SK-015655, add the two holes for the location of molded inserts and trim periphery.

9.0 Layup (attach angles and caps)

9.1 Remove polyimide prepreg (35-512) from refrigeration and record roll number, batch number, and verification date, and record in laboratory notebook.

> Note: Allow the material to come to room temperature before removal from sealed container.

9.2 Lay up ± 45 degree plies (6) on a flat plate wide enough to form angle, debulk as necessary, include layup for caps (5-ply laminate).

9.3 Rough trim angle layup to size.

9.4 Keeping the correct ± 45 degree orientation, apply the 6 plies to the angle tool, and add thermocouples.

9.5 Lay up a 7-ply laminate, 5 by 8 inches for flexural test coupons and add thermocouples.

9.6 Apply parting fabric, bleeder cloth, and vacuum bag.

> Note: Include angles, caps, and test coupons in the same vacuum bag.

9.7 Add static vacuum gauge, vacuum line connections, and check as in 6.12, above.

10.0 Same as SK-015653 (5.1 through 5.7).

11.0 Postcure (angles and caps)

11.1 Place angles back on the tool, and the caps and test coupons on a flat plate, restrain the angles caps and coupons, and add the necessary ther thermocouples.

11.2 Place tool and parts in the oven and connect thermocouple connections.

11.3 Prepare cure card (Form 2972Y) and record serial number in laboratory notebook.

11.4 Secure oven and post cure angles, caps, and coupons the same as in SK015653 (6.4 a through g).

3

11.5 Remove angles, caps, and coupons from the tool and clean flashings, etc., and visually inspect.

11.6 Trim one leg of angle to drawing dimension.

11.7 Submit test coupons on Form 2906-C to Process Control Laboratory, D/098, for test.

11.8 Verify process control laboratory report.

12.0 Layup (edge inserts)

12.1 Same as 9.1 above, except use 35-503 prepreg.

12.2 Lay up unidirectional plies on a flat aluminum plate in the following order: Lay up three prepreg and one dry 181 cloth alternately until a total of 40 plies have been laid up; then add two extra plies of prepreg; add thermocouples.

12.3 Apply parting fabric, honeycomb and bleeder cloth, and vacuum bag.

12.4 Add static vacuum gauge, vacuum line connections, and check as in 6.12 above.

13.0 Curing (edge inserts)

13.1 Place entire bagged unit into the oven.

13.2 Connect vacuum lines and thermocouple connections.

13.3 Recheck for vacuum loss as in 6.12 above.

13.4 Prepare cure card (Form 2972Y and record serial number in laboratory notebook.

13.5 Secure oven and cure at the following schedule:

a. Apply full vacuum pressure.

b. Raise temperature to 300 F ± 10 F and hold for a minimum of 8 hours.

c. Cool to 150 F under full vacuum pressure before removal from oven.

13.6 Disconnect thermocouple leads, remove vacuum bag, and bleeder cloths, etc.

13.7 Reconnect thermocouple leads, and rebag without the honeycomb.

13.8 Continue cure in the autoclave according to the following schedule:

a. Apply full vacuum pressure and 90 psig positive pressure.

b. Raise temperature to 350 F ± 10 F and hold for 2 1/2 hours (minimum).

c. Cool to 150 F under full vacuum and 90 psig positive pressure before removal from the autoclave.

d. Disconnect thermocouple leads; remove vacuum bag and bleeder cloth.

e. Visually inspect.

14.0 Post cure (edge inserts)

14.1 Position precured 6 by 43 inch part in the press on a release-coated 1 inch aluminum plate with 375-inch thick longitudinal members (aluminum) the full length part (43 inches); add a second release-coated 1 inch aluminum plate to the top of the part, and add thermocouples.

14.2 Prepare cure card (Form 2972Y) and record serial number in laboratory notebook.

14.3 Close the press and post-cure in accordance with the following schedule.

a. Raise the temperature to 550 F ± 10 F and hold for 4 hours.

b. Cool to 150 F while still under pressure.

14.4 Cut part into 4 pieces to match drawing dimensions and add tool holes, using SK-015654, SK-015655, or SK-015656.

15.0 Trim and prime (core)

15.1 Cut core to size and add insert holes using tool SK-015655.

15.2 Same as SK-015653 (8.2 through 8.4).

16.0 Layup (subassembly A of skins, core, molded inserts, and edge inserts)

16.1 Part preparation

a. Core (prepared in detail section).

b. Face sheets (light-sand with Scotch-Brite type A and remove dust by washing surface with MEK followed by BR-34-2 thinner, and wipe dry with clean lint-free gauze).

c. Edge inserts (light sand with 120 to 180 gritpaper and then clean with MEK).

d. Molded inserts (same as (c) above).

16.2 Remove adhesive (FM-34, 0.135 psf) from refrigeration and allow to come to room temperature before removal from sealed container. Record roll number, batch number, and verification date.

16.3 Apply adhesive to one skin and locate on tool (SK-015656.

16.4 Position molded inserts, apply FM-29 foam tape to area of core bond to molded and edge inserts, add core and edge inserts.

16.5 Apply adhesive to second skin and locate on subassembly.

16.6 Lay up required test coupons (lapshear and flatwise tension — see 6.10 above for lap shear configuration) 6 by 6 inches 0.020 inch 2024 T6-Al bonded to aluminum honeycomb core, MRO 170-27 Type 1 Class 0/16 0.003 inch for flatwise tension.

16.7 Add thermocouples as required to both test coupons and subassembly.

16.8 Apply parting fabric bleeder cloth and vacuum bag material to both the assembly and test coupons.

Note: Include subassembly and test coupons in one vacuum bag.

16.9 Add static vacuum gauge, vacuum line connections, and check in accordance with 6.12 above.

17.0 Curing (Subassembly A)

17.1 Same as SK-015653 (10.1 through 10.7)

18.0 Post cure (Subassembly A)

18.1 Same as SK-015653 (11.1 through 11.7)

19.0 Lay up (main assembly, angles, caps, and titanium double to Subassembly A

19.1 Part preparation

a. Attach angles and caps, light sand with 120 to 180 paper, and clean with MEK.

b. For Subassembly A, in the area of bonding, follow procedure 16.1b above.

19.2 Remove adhesive (FM-34, 0.135 psf) from refrigeration, and allow to come to room temperature before removal from sealed container. Record roll number, batch number, and verification date.

19.3 Apply adhesive to attach angles, caps and titanium doubles, and apply FM-29 foam tape to exposed core edge.

19.4 Position attach angles and caps and titanium doublers to Subassembly A, add required thermocouples.

19.5 Using bars SK-015658 and rubber pad tooling, clamp in position.

20.0 Curing (main assembly)

20.1 Same as SK-015653 (10.1 through 10.7)

21.0 Post cure (main assembly)

21.1 Spar main assembly will be post cured on box beam final assembly. However, test coupons will be post cured by themselves the same as SK-015653 (11.1 through 11.7).

22.0 Final inspection